Thermal Performance and Reliability of Liquid Metal Pastes With Solid Metal Particles as Thermal Interface Materials for the Cooling of Computing Electronics

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Abstract
In recent years, we have developed a series of gallium-based liquid metal paste containing metal particles (LMPMPs) to enhance thermal performance and control BLT of the prepared liquid metal composites as thermal interface materials (TIMs) synthesized via in-situ introducing gallium oxide into the liquid metal alloys. In our recent research, we intensively investigated thermal properties of different primary liquid metal alloys containing low contents of solid metal additives and have extensively run the reliability tests such as power cycling, thermal cycling and aging tests on the synthesized LMPMPs. In this paper, we will report thermal properties of the synthesized LMPMPs based on different liquid metal alloys and reliability test that mainly focuses on power cycling test by monitoring change of thermal resistance of LMPMPs over time by using a house-made power cycler. We have found significant influences of power density, bond line thickness, different liquid metal alloys on thermal properties and reliability of LMPMPs. We also conducted thermal aging test on the Cu-LMPMPs-Cu sandwich specimens at 85°C in ambient atmosphere to study thermal conductivity deterioration of LMPMPs over time, measured by laser flash analyzer using layered structure method. Highly accelerated stress test (HAST) with a profile of 110°C, 85RH%, 264 hours was used to study the decomposition of liquid metal alloys. Thermal cycling test with a profile of -40 to 125°C was employed to investigate the pump out of LMPMPs. It has been proven that LMPMPs have much better thermal performances of polymer-based thermal grease.

Key words: Liquid metal, thermal interface materials, thermal conductivity, resistance, highly accelerated stress test (HAST), power cycling and thermal cycling tests.

I. Introduction
With the down-size and increase of power density of microprocessors, effective and quick dissipation of heat generated from high power chips is crucial for the extension of long-term reliability or life-span of electronics package and devices [1], [2]. Gallium-based liquid metal (LM) with fairly high thermal conductivity, low interfacial resistance and fluidic characteristics has been widely considered an ideal thermal interface material capable of effectively dissipating heat from heating sources like microprocessors, i.e. CPU, GPU, MCM, to heat sink in high power density electronics, especially in high performance computing and gaming electronics [2], [3]. Ga-based liquid metal alloys, including the alloys of EGaIn, Galinstan, GaInSnZn etc. have been proven to offer both low interfacial resistance and fairly high conductivity; and have also been widely used as liquid metal thermal interface materials[4], [5]. Although LMAs could provide both low interfacial resistance and high conductivity, they have intrinsic drawbacks like various reliability issues including: corrosion/oxidation, intermetallic formation, drip-out, dewetting, and migration[6], [7], [8]. Due to the active reaction of gallium, these mechanisms will continue to deteriorate the interface, resulting in a thermally related catastrophic failure of the
actual electronic component during operating applications. Additionally, the high surface tension of Ga-based LMAs lead to poor wettability and adhesion to different surfaces of electronic components such as bare Cu, Si, Ni etc., which may cause extreme challenge when conducting LMAs by traditional deposition process. Several research groups demonstrated various methods to mitigate these problems [8], [9], [10]. For examples, the oxidation of LMAs can be minimized by a hermetic seal, and the formation of intermetallic compounds can be prevented by applying a diffusion barrier such as Ni, Cr, W surface finish heat sink or spreader. Another mitigation method is Ga-based liquid metal pastes containing high content of solid metal particles with high viscosity for thermal and electrical connections was first disclosed by Thomas Tolbear et. al. [3]. Moreover, a number of research groups recently reported that highly thermally conductive fillers, such as graphene [11], diamond [12], copper [13], [14], tungsten [15] and boron nitride [16], are often blended into liquid metals to improve the adhesion and wettability of liquid metal composites, as well as to further enhance their thermal performance in the thermal management of high-power electronics. However, little is known about influence of low content of metal particle additives on thermal properties, wetting/adhesion, deposition process performance of liquid metals. To improve wettability and adhesion of LMAs on various components in the electronics, thermal and process performance, we have developed a series of gallium-based liquid metal paste containing low content of solid metal particle additives (LMPMPs) to enhance thermal performance and control BLT of the prepared liquid metal composites. This new material consists of thermal interface materials (TIMs) synthesized via in-situ introducing gallium oxide into the liquid metal alloys [17], [18], which offers better thermal and printing performance. In addition, we have also developed a series of liquid metal alloys containing organic compound additives that has been proven for excellent dispensing performance due to enhancement of wettability and adhesion to bare Cu PCB and OSP copper coupons [19]. In this paper, we will report thermal properties of different primary liquid metal alloys containing low contents of solid metal additives and their reliability results obtained from power cycling, thermal cycling and aging tests on the synthesized LMPMPs.

II. Materials and Experiment

A. Materials and Preparation of LMPMPs
Gallium eutectic (300E), GaInSn eutectic (51E), and GaInSnZn (Ind2N) were produced by Indium Corporation and used as received. Metal particles were purchased from Alfa-Aesar Inc and used as received. A general process is described as follows: Liquid metal paste containing metal additives were prepared from mixing liquid metal alloys with metal particles. Then in-situ introducing gallium oxide by stirring it with mechanical crossed blade impeller for approximately one hour until a large volume of LMPMPs are produced. Likewise, we have prepared three series of liquid metal paste based on 300E, 51E and Ind2N with/without metal additives.

B. Measurement of thermal conductivity and resistance
Thermal tester, TIMA5, made by Nano test, Germany was utilized to measure thermal conductivity and resistance of LMPs based on ASTM D-5470-18 at 50°C and in different pressures. House-made thermal tester, based on ASTM D-5470, with a Picolog-TC08 data logger (Pico Technology) to collect gradient temperatures on hot/cold copper meter bar was used to measure and monitor thermal resistance of LMPs over time at around 72°C.

C. Power cycling test
A house-made power cycler with a relay switch, GRT8-S1, to control power "On” and “Off” with 50W input power, 1hr on/off was used to run power cycling test on LMPMPs. Thermal resistance, Rth, is calculated from the gradient temperatures recorded by the Picolog-TC08 (Pico Technology) or GL240 data logger (Graphitec America, Inc.) on the cold and hot Cu meter bars when the power is on.

D. DSC/TGA of LMA and LMPMPs
DSC (TA, Q2000) and SDT (TA, Q600) were used to measure the crystalizing and melting temperatures of liquid metal alloys and liquid metal pastes with and without solid liquid metal particle additives, with the in the heating and cooling rate of 10°C in the nitrogen atmosphere. Melting temperatures of the LM, LMPs and LMPMPs were determined on the second heating run.

E. HAST, Thermal aging tests, and TCT
Thermal cycling test chamber (Tenny-10, Tenny Company) with a profile of -40°C to 125°C, dwell time of 10min at low and high temperature, highly accelerated stress test system (EHS-222M, ESPEC) with a profile of 110°C, 85RH%/0.122MPa, 264hours were used to run thermal cycling and stress tests on LMPMPs, respectively. Thermal aging test was conducted using a stationary oven at 85°C, followed by measuring thermal conductivity of LMPMPs to determine the degradation of LMPMPs over time at the elevated temperature.

F. Measurement of Viscosity
Viscosity of the LMPS and LMPMPs were measured using a cone/plate viscometer (AMETECK Brookfield...
viscometer, DV2T) with different speeds (10rpm and 5rpm) and 25°C.

III. Results and Discussion

A. Viscosity measurement and DSC test

Reported within our previous paper, liquid metal paste has much higher viscosity than that of liquid metals, and phase separation was observed due to the gallium oxide dispersed in the matrix of liquid metals [17]. In the present research, we have investigated the influence of metal additives on viscosity of liquid metal pastes with and/or without the additives. Figure 1 presents viscosity of liquid metal pastes with and without solid metal additives measured at 25°C. In the meantime, it became evident that LMPs and LMPMPs show “shear thinning” behavior occurring with increase of shear rate which perfectly match power law model, as shown in Figure 1 [20]. It is obvious that the existence of Ga$_2$O$_3$ in LMP and LMPMP lead to higher viscosity values than that of LM51E. A three-step shear test on liquid metal, LMP and LMPMP was performed in this research. The results obtained through this research indicate that viscosity of LMPMPs look more stable than LMPs as it could not fully recover viscosity values when the shear rate resume to 19 (1/s) from 39 (1/s) and also scatter slightly at each step, as shown in Figure 2. On the same note, this research implied that metal particles play a specific role in the deforming of microstructure of LMPMPs test. However, LM 51E, show the similar stability of viscosity during a three-step shear test.

DSC results reveal that LMPs and LMPMPs have significant influence on phase transitions, especially, crystallizing temperature on the cooling curve at which only shows single phase transition or crystallizing temperature at -26.2°C, whereas primary liquid metal alloy 51E displayed two crystallizing temperatures at -74°C and -47°C, respectively, as shown in Figure 3. Although LMPs and LMPMPs have the similar melting temperatures to the primary liquid metals. It is obvious that the existence of gallium oxide (Ga$_2$O$_3$) significantly affects the nucleation and the crystal growth of liquid metal alloys, which lead to high crystallizing temperature.
B. Measurement of Thermal Conductivity and Rth

Thermal conductivity and resistance of liquid metal and liquid metal pastes (LMPs and PMPMPs) have been measured by using a thermal tester (TIMA5, Nanotest) based on the methodology (ASTM D5470) with CuCrN-R3-23A test heads at 50°C. Intrinsic thermal conductivity and interfacial resistance of the LM, LMPs and LMPMPs were determined from the plot of thermal resistance vs. bondline thickness. It is known that total effective thermal resistance, Rth_{eff}, can be determined according to Eq. (1).

\[
R_{th_{eff}} = \frac{\Delta T}{Q} \quad (1)
\]

\[
R_{th_{eff}} = R_{th_{bulk}} + R_{th_{C}} \quad (2)
\]

\[
R_{th_{eff}} = \frac{1}{\lambda_{bulk}A} \times BLT + R_{th_{C}} \quad (3)
\]

where Rth_{eff} is total thermal resistance of the bulk and contact resistances that is expressed in Eq. (2). Intrinsic thermal conductivity of LMPs and LMAs can be calculated from the reciprocal of plot slope of thermal impedance vs. BLT, whereas the incept is interfacial resistance of LMPs and LMAs according to Eq. (3), respectively. For an instance, Figure 4 presents the plot of thermal resistance of liquid metal alloys (51E) vs. bondline thickness (BLT), and determination of intrinsic thermal conductivity and interfacial resistance of 51E. Intrinsic thermal conductivity and resistance of all tested samples, LMPs, LMPMPs and LMAs, are summarized in Table I.

Figure 4. Plot of thermal resistance vs BLT of liquid metal eutectics (51E) at 50°C for usage of the calculation of intrinsic conductivity and interfacial resistance.

Although thermal conductivity of LMPs are fairly lower than that of primary liquid metals, thermal resistance of LMPs are quite similar or comparative to the LMAs.

Table I. Intrinsic Conductivity and Interfacial Resistance at 50°C

<table>
<thead>
<tr>
<th>Code No.</th>
<th>Name</th>
<th>TC, W/m K</th>
<th>Rth, K/W</th>
<th>θ, K mm²/W</th>
<th>Coefficient, R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>LA390381</td>
<td>LM-51E</td>
<td>24.22</td>
<td>0.018</td>
<td>9.40</td>
<td>0.9851</td>
</tr>
<tr>
<td>LA378966</td>
<td>LM-300E</td>
<td>24.27</td>
<td>0.034</td>
<td>16.92</td>
<td>0.9757</td>
</tr>
<tr>
<td>LA253256</td>
<td>Indalloy1N</td>
<td>27.897</td>
<td>0.014</td>
<td>9.54</td>
<td>0.9722</td>
</tr>
<tr>
<td>LA258884</td>
<td>Indalloy2N</td>
<td>27.100</td>
<td>0.028</td>
<td>13.96</td>
<td>0.9819</td>
</tr>
<tr>
<td>897-92-1</td>
<td>LMP92-1</td>
<td>9.470</td>
<td>0.003</td>
<td>1.47</td>
<td>0.9072</td>
</tr>
<tr>
<td>897-92-2</td>
<td>LMP92-2</td>
<td>11.779</td>
<td>0.008</td>
<td>3.94</td>
<td>0.9406</td>
</tr>
<tr>
<td>897-92-3</td>
<td>LMP92-3</td>
<td>16.611</td>
<td>0.023</td>
<td>12.68</td>
<td>0.9007</td>
</tr>
<tr>
<td>897-82-6</td>
<td>LMP82-6</td>
<td>13.528</td>
<td>0.013</td>
<td>6.64</td>
<td>0.9770</td>
</tr>
<tr>
<td>897-82-7</td>
<td>LMP82-7</td>
<td>11.717</td>
<td>0.006</td>
<td>3.11</td>
<td>0.9919</td>
</tr>
<tr>
<td>897-83-3</td>
<td>LMP83-3</td>
<td>25.342</td>
<td>0.029</td>
<td>14.46</td>
<td>0.9456</td>
</tr>
</tbody>
</table>

However, we found in this research that LMPs with and without additives have shown better wettability and adhesion than that of LMAs on different substances. This result can be explained since LMAs have much higher surface tension, which leads to poor wetting on Cu, Glass, and Silicon [17], which might result in increasing the interfacial resistance although LMAs has higher thermal conductivity than that of LMPs. In the meantime, the high surface tension makes liquid metals to become a tough material to handle by traditional deposition process such as printing and dispensing.

C. Power Cycling Test

Figure 5 (a, b). Rth of LMP82-6 as a function of different input power of 60, 80, 90, 100, 120W with 1hr on/off: a. Rth at time 0; b. Rth within 11 cycles.
In order to evaluate the reliability of liquid metal pastes, a power cycling tester was made in house to run the power cycling test on LMPs at the input power of 60W, 80W, 90W, 100W, 120W and 1hr on/off per cycle. Figure 5a presents Rth values of LMP82-6 with the sample loading amount of 0.5g as a function of input power from 60 to 120W at time 0. It is very interesting that Rth values of LMP82-6 decrease with increase of the input powers except for 90W as the preliminary data show. With increase of power cycles, thermal resistance of LMP82-6 remain the same values as time 0 as the input power is below 100W, but thermal resistance dramatically increase after 2cycles of 1hr on/off with the input power is 120W, as shown in Fig. 5b.

![Image](image1.png)

**Figure 5.** Rth vs Power Cycles (60W of Input Power)

- **a. 60W input power**
- **b. 120W input power**

Figure 6. Rth of LMP82-6 as a function of sample loading with input power 60W and 120W, 1hr on/off: a. 60W; b. 120W.

Power cycling tests on different amount of the loading samples using 60W and 120W of input powers, 1hr on/off was conducted to investigate the influence of BLT on thermal resistance of LMPs and LMMPMs. As you can see in Fig. 6a, that Rth of LMMPMs significantly increase with the increase of the loading amounts of samples, and thermal resistance values remain the quite same as time 0 within 10cycles except for the sample loading amount of 2.0 gram, as shown in Fig. 6a. When the input power is 120W, thermal resistance values increase dramatically with an increase of the sample loading amount from 0.5gram to 1.0gram, and then become very similar as the sample loading amount increases from 1.0 to 2.0gram, as shown in Fig. 6b. With 120W of input power and 1hr on/off, thermal resistance value dramatically increases within 5cycles, and then remains consistent regardless of the loading amounts of samples, as shown in Fig.7. It is obvious that BLT significant affect Rth of LMPs.

![Image](image2.png)

**Figure 7.** Rth of LMP82-6 as a function of power cycles with input power of 120W, 1hr on/off and different loading of the samples.

In the meantime, we investigated the influence of different liquid metal alloys on thermal properties of LMPs. Founding from this investigation is that LMPs based on different primary liquid metal alloys yield different thermal resistance when the sample loading amount is 1gram; moreover, there were no significant difference except for 897-92-1 and 897-82-6, as show in Fig. 8.

![Image](image3.png)

**Figure 8.** Rth of LMPs made from different liquid metal alloys as a function of power cycling with input power of 60W, 1hr on/off.
For the evaluation of hundreds-of-cycle-long data monitoring, the in-house power cyclers with 1hr on/off multi-power input and GL240 datalogging features was used to record the temperature gradient of LMPs. For an example, we have run a test on LMP of 897-82-6 with loading sample of 1.0gram, 60W input power, 1hr on/off, and found no change of thermal resistance after 475cycles, as shown in Fig. 9 (Unfortunately we could not record temperature gradient at that moment due to the software issues with GL240 data logger).

When the input power is 100W, Rth remains the same value of 0.02K/W after the totally accumulated number of power cycling is at 100cycles, as shown in Fig. 10. No significant change of thermal resistance was observed, yet obvious high power thermal resistance was observed when the sample loading was at 1gram or greater, due to the leakage of liquid metal during test.

Overall, LMP82-6 has very good reliability in the low power condition like 60w of input power, at which there is no change of thermal resistance after 475cycles, as shown in Fig. 9.

To further investigate the influence of high power and BLT on thermal resistance of LMPMPs, we have conducted a test on 897-82-6 using a power cycler with 120W of input power, 1hr on/off and the different loading amounts of samples. With input power of 120W and loading sample of 0.5g, thermal resistance increases significantly within 5cycles at first, and gradually increases up to 0.012K/W after 100cycles and then rises over 0.012K/W after 175cycles, as shown in Fig.11a. There was the pump-out phenomenon of the liquid metal observed at the beginning of test, which is major reason for the dramtical increas of thermal resistance values within 5cycles, but thermal resistance remains steady value as low as 0.013K/W after 200cycles until it completes at 526cycles.

With an increase of the loading amount of sample, thermal resistance of LMP82-6 is slightly higher than that of the low loading amount of sample, as shown in...
Fig 11b. It implies that thin BLT, as a small amount of sample was loaded, would be helpful to minimizing or reducing thermal resistance of LMP82-6 as is tested. It remains thermal resistance values as the same as $R_{th}$ at 100cycles. However, the loading amount of sample doesn’t affect the trend of change of thermal resistance of LMP82-6. 

![Thermal Resistance Plot - Tester 1](image1)

a. 0.5g of sample, 120W of input power, 1hr on/off

b. 1.0g of sample, 120W of input power, 1hr on/off

Figure 11 (a, b). $R_{th}$ of LMP82-6 as a function of power cycling with 120W, 1hr on/off and 0.5 and 1.0gram of sample.

We have also investigated the influence of time for power on/off on thermal resistance of LMPs, and found that short time of the on/off profile would have influence on thermal resistance of LMPs. For example, when the power cycling test with a profile of 60W of input power, 15min on/off, thermal resistance values of LMP82-6 is much higher than that of LMP82-6 tested with a profile of 1hr on/off time, as shown Fig 12 (a, b). In the meantime, there was pump-out phenomenon of liquid metal observed during test as the power cycling test with 60W, 15min on/off was applied, whereas no pump-out appeared as the power cycling with 1hr on/off was applied. It is interesting to note that thermal resistance values had no significant change of LMP82-6 over time within 100cycles during test though the pump-out was observed when short-time of power on/off was applied. It is perhaps because of low input power of 60W used in this test.

![Thermal Resistance Plot - Tester 1](image2)

a. 60W, 60min on/off

b. 60W, 15min on/off

Figure 12 (a, b). $R_{th}$ of LMP82-6 as a function of power cycling with input power of 60W, 15min and 15min on/off and 1gram of sample.

**D. HAST, TCT and High Temperature Aging**

Microlubrol Sylcap284-F silicone elastomer encapsulant was used to encapsulate liquid metals 51E and LMP82-6 in bare Cu lids, as shown in Fig. 13. HAST with a profile of 110°C, 85RH%/0.122MPa, 264hours (Similar to JESD-22-A110-B), was used to evaluate the reliability and stability of LMP82-6 and LMAs, but is not active test device used in this test. After 264hours in HAST chamber, the pump-out of 51E and LMP82-6 were observed, but still covered underneath PDMS encapsulant and the samples remain the very similar colors as the beginning of test though it is no longer shining. There is no degradation of the encapsulants observed after HAST for 264hrs.
Another set of the samples of 51E and LMP82-6 were encapsulated with Microlubrol Sylcap284-F silicone elastomer by curing at ambient temperature for 48 hours, followed by post-treatment at 85°C, in 27In Hg vacuum for 12hrs and then holding temperature at 85°C under N₂ in a vacuum oven for a week. HAST with the profile of 110°C, 85RH%, 264hrs/0.122MPa on the prepared samples was conducted. There was no leakage observed during the post-treatment after the curing of encapsulants, but it was obvious that the colors of 51E and LMP82-6 changed, as shown in Fig.14c. It was perhaps due to the oxidation of gallium because oxygen would permeate through PDMS very well. Pump-out behavior of both LMP and 51E was observed after HAST, as shown in Fig. 14d.

High temperature aging test on liquid metal and liquid metal paste held between two copper coupons was conducted in stationary oven at 85°C, followed by measurement of thermal conductivity using LFA. Aging test conducted over time indicates that thermal
conductivity value of LMs decrease obviously compared to liquid metal alloys, as shown in Fig. 15. It is obvious that the existence of Ga₂O₃ might cause porous structure in the pastes that enable oxygen diffuse into the paste faster, which results in the severe oxidation of gallium in the pastes between two copper coupons. Liquid metal alloys, however, could form the fresh peripheral skin of Ga₂O₃ between two copper coupons that would protect LMs from the further oxidation during aging test. It implies that it is necessary to seal liquid metal and liquid metal pastes so as to avoid the oxidation of Gallium in the liquid metal paste and liquid metals during test and operations.

![Figure 15](image1.png)

Figure 15. Thermal conductivity of liquid metal and liquid metal pastes measured by LFA after aging test at 85°C.

Additionally, thermal cycling test with a profile of -40°C to 125°C, 10min of dwell time, is used to evaluate reliability of LMPs and LMPMPs and in progress. There is no pump-out observed after 700cycles of -40°C to 125°C, as shown in Fig 16.

![Figure 16](image2.png)

Figure 16. Optical image of LMPMPs (LMP82-6) using a profile of -40°C to 125°C, 10min of dwell time at low and high temperature.

Conclusion
A series of liquid metal pastes with and/or without metal additives (LMPs & LMPMPs) have been synthesized via in-situ oxidation of primary liquid metal alloys. LMP and LMPMPs have significantly higher viscosity than that of primary LMs. Addition of metal additives to LMPs would stabilize microstructure of liquid metal pastes. Though thermal conductivity values of LMPs and LMPMPs are fairly lower than that of LMs, thermal resistance and interfacial resistance are quite similar to the primary LMs. LMP and LMPMPs with thin BLT shows much lower thermal resistance and good reliability. BLT between 70micron and 150micron has no significant difference of thermal resistance, but BLT up to 200micron has significant influence on thermal resistance of LMPs and LMPMPs. Despite thermal resistance of LMPs and LMPMPs increases with increasing input power from 60W to 120W and shorter cycling time, they are not significant different as data analysis indicates. Since LMPs and LMPMPs have high viscosity, good wetting and adhesion on substrate or test head, it could achieve very thin BLT at around 50nm or less, which would benefit thermal performance or transfer heat energy quickly. Power cycling test with a profile of input power of 120W or less, 1hr on/off reveals that LMPMPs with thin BLT has excellent reliability as it was even tested in ambient atmosphere. HAST test show that LMP/LMPMPs have less pump-out issues, but is not significant. High temperature aging test indicates that thermal conductivity of LMPMPs deteriorates due to the further oxidation of Ga in the LMPs. In real application, it is necessary to seal LMPs within components with a sealant or dam in electronics devices. Thermal cycling test with a profile of -40°C to 125°C, 10min of dwell time is in progress.

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