ORIGINAL ARTICLE

Conductive adhesive with transient liquid-phase sintering technology for high-power device applications

Yong-Sung Eom 🔘 | Keon-Soo Jang | Ji-Hye Son | Hyun-Cheol Bae | Kwang-Seong Choi

ICT Materials & Components
Research Laboratory, Electronics and
Telecommunications Research Institute,
Daejeon, Rep. of Korea

Correspondence
Yong-Sung Eom, ICT Materials &
Components Research Laboratory,
Electronics and Telecommunications
Research Institute, Daejeon, Rep. of Korea.
Email: yseom@etri.re.kr

A highly reliable conductive adhesive obtained by transient liquid-phase sintering (TLPS) technologies is studied for use in high-power device packaging. TLPS involves the low-temperature reaction of a low-melting metal or alloy with a high-melting metal or alloy to form a reacted metal matrix. For a TLPS material (consisting of Ag-coated Cu, a Sn96.5-Ag3.0-Cu0.5 solder, and a volatile fluxing resin) used herein, the melting temperature of the metal matrix exceeds the bonding temperature. After bonding of the TLPS material, a unique melting peak of TLPS is observed at 356 °C, consistent with the transient behavior of $Ag_3Sn + Cu_6Sn_5 \rightarrow$ liquid $+ Cu_3Sn$ reported by the National Institute of Standards and Technology. The TLPS material shows superior thermal conductivity as compared with other commercially available Ag pastes under the same specimen preparation conditions. In conclusion, the TLPS material can be a promising candidate for a highly reliable conductive adhesive in power device packaging because remelting of the SAC305 solder, which is widely used in conventional power modules, is not observed.

KEYWORDS
conductive adhesive, device packaging, high reliability, power module, transient liquid-phase sintering

1 | INTRODUCTION

Power semiconductors are important electrical components that convert AC into DC or voltage changes in electrical vehicles, power transmission units, industrial machinery, and consumer electronics. One of the important issues in industrial applications is the need for power modules to ensure safe operation of machines; therefore, these modules must show excellent durability and reliability. Conventional power modules, as shown in Figure 1, are interconnected to a metallized substrate using a conductive material such as a solder and mounted on a heat sink using thermal grease.

Currently used modules require a high-temperature operating power module for performance improvement because

the conductive material between the power device and the substrate during heating and thermal cycles must withstand degradation of the components, such as crack formation or melting. Power modules based on GaN or SiC can be applied to high-temperature (>200 °C) operations, and conductive materials for interconnections should be identified by considering the reliability of the material [1–3]. SAC305, a well-known Sn-based lead-free solder with a melting temperature of 220 °C, is widely used in conventional power modules, but is unsuitable for high-temperature applications in power device packaging. Other alternatives such as metal-based adhesives, including sintered Ag or Au/Sn solders, are difficult to apply in electrical vehicles owing to their high cost and low adhesion strength.
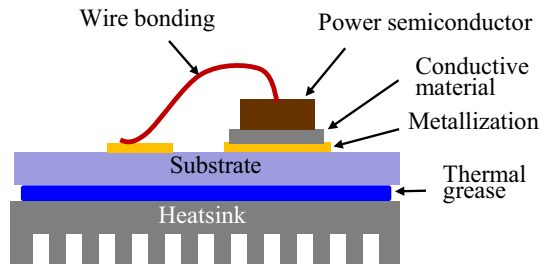
FIGURE 1 Conventional power module structure

In this study, an interconnection material based obtained by transient liquid-phase sintering (TLPS) with copper and the SAC305 solder is investigated. Figure 2 shows the mechanism of thermal and electrical conduction for a TLPS material consisting of Cu flakes, a solder, and a volatile fluxing resin. Previous studies have focused on the mechanism of interconnection between Cu and a solder based on an epoxy matrix [4–11]. As shown in Figure 2A, the TLPS material composed of Cu and the solder dispersed in the volatile fluxing resin provides an interconnection between the power semiconductor and the substrate. As the temperature increases, the activated volatile fluxing resin removes oxides on the surfaces of the metal particles; as a result, the solder is wetted and interconnected between the Cu particles at the melting temperature of the solder. The volatile fluxing resin continuously evaporates, whereas Cu and the solder are converted into a TLPS material during the bonding process. Finally, for high-temperature reliability, interconnections between the power semiconductors and the substrate are completed using a TLPS material with a minimal organic matrix.

2 | MATERIALS AND EXPERIMENT

2.1 | Materials

For the TLPS paste, three base resins and two reductants were selected to remove the oxides on the surfaces of the metal fillers, as shown in Table 1. Six types of volatile fluxing resins were prepared: a base resin (100) and 25 phr of a reductant. The mixing weight percent ratio of the volatile fluxing resin, Cu flakes (Ag-coated Cu), and solder (Sn/3.0Ag/0.5Cu) was determined as 18:45:37 to obtain the optimum viscosity for screen printing. The boiling temperatures of all the chemical components used are shown in Table 2. Figure 3 shows a scanning electron microscope (SEM) image of Ag-coated Cu with an average diameter of 5 μm and Sn/3.0Ag/0.5Cu solder type 7. The diameter range is 2 μm to11 μm. The volatile fluxing resin was prepared using a roll-mill machine because at room temperature, the reductants were in the solid state and the base resins were in the liquid state.

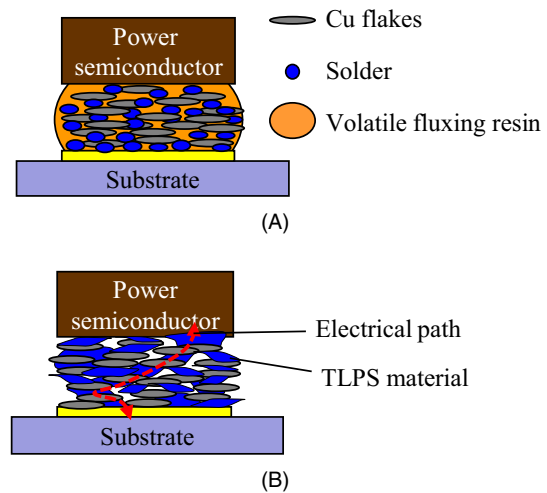To compare the thermal conductivity between the TLPS material and the commercialized Ag pastes, three

FIGURE 2 Schematics of conduction mechanism with transient liquid-phase sintering (TLPS) material: (A) Before and (B) after the bonding process

TABLE 1 Composition of TLPS materials

Material	Component	Content (wt.%)
Volatile fluxing resin (base resin (100)/reductant (25 phr))	A/D	18
	B/D	
	C/D	
	A/E	
	B/E	
	C/E	
Cu flakes	Ag-coated Cu	45
Solder	Sn/3.0Ag/0.5Cu	37

TABLE 2 Boiling temperature of chemical components for TLPS materials

Component	Boiling temperature (°C)
A	185
B	242
C	198
D	286
E	200

Ag sintering pastes—ASP 295-79P2(D1) from Heraus, TS 3332-LD from Tanaka, and Atrox D800HT2V from Alpha Advanced Materials—were applied, as shown in Table 3.

2.2 | Experiment

The weight change of the TLPS paste was measured under isothermal conditions in a nitrogen environment using a thermogravimetric analyzer (TGA) from TA Instruments.
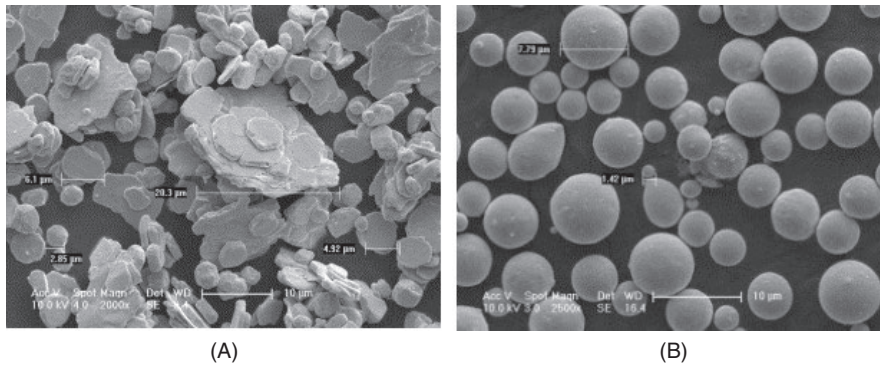
FIGURE 3 SEM images of metal fillers: (A) Ag-coated Cu and (B) Sn/3.0Ag/0.5Cu solder type 7

TABLE 3 Catalog of commercialized Ag sintering pastes

Manufacturer	Heraus	Tanaka	Alpha advanced materials
Model	ASP 295-79P2(D1)	TS 3332-LD	ATROX D800HT2V
Filler	Ag	Ag	Ag
Processing conditions	160 °C/30 m + 230 °C/60 m	120 °C/30 m + 200 °C/30 m	150 °C/30 m + 200 °C/90 m
Thermal conductivity (W/m.K)	>100	24.2	>130

Screen printing and sintering were conducted to measure the volume resistivity of the sintered TLPS material. TLPS paste was printed on a high-resistivity silicon substrate at room temperature with a 0.1-mm-thick metal mask, and sintered in a nitrogen environment at 240 °C for 10 minutes using an SMT machine (SK-5000, Sanyo) as shown in Figure 4. The volume resistivity was measured by a four-point probe method using a FPP-RS8 equipment. Differential scanning calorimetry (DSC) was carried out at a heating rate of 10 °C/min, from 25 °C to 450 °C, for thermal analysis. The viscosity of the paste was measured at 10 rpm using a viscometer (HBDV-II, Brookfield).

For thermal conductivity measurements, conductive paste was printed on a copper plate (12.7 mm in diameter and 1.45 mm in thickness), followed by sintering in an oven under a nitrogen environment, as shown in Figure 5. The thermal conductivity was measured by a laser flash method using a LFA447 equipment. The cross-section and metallic composition of the sintered TLPS material were analyzed by scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX).

The shear strength of the conductive material was measured, as shown in Figure 6. For the sample preparation, a silicon dummy device (2 mm × 2 mm × 0.6 mm) was placed on a silicon or ceramic substrate and sintered using a conductive material. The shear strength was measured by a die-shear test at room temperature using a Dage 4000 equipment.
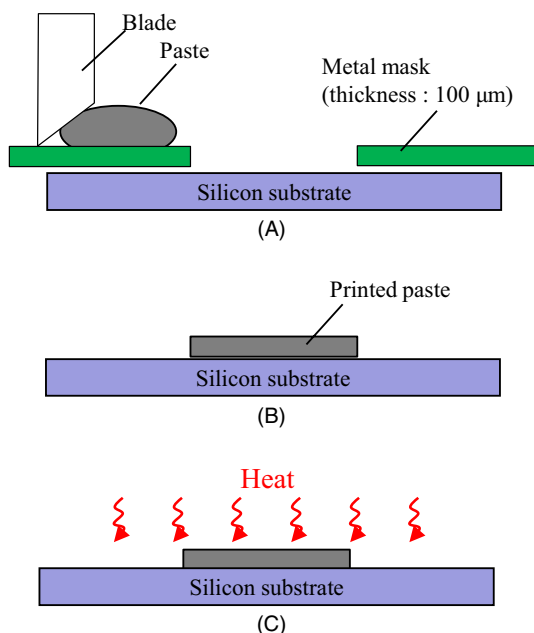
3 | RESULTS AND DISCUSSION

For a power semiconductor operated at high temperatures, the TLPS-based conductive material must satisfy

FIGURE 4 Schematic of screen printing and transient liquid-phase sintering (TLPS): (A) Alignment of metal mask with 0.1 mm thickness on a high-resistivity silicon substrate by (B) screen printing process TLPS paste, and (C) sintering in a nitrogen environment
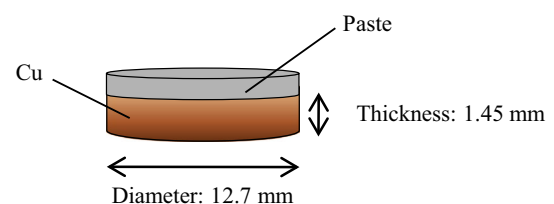

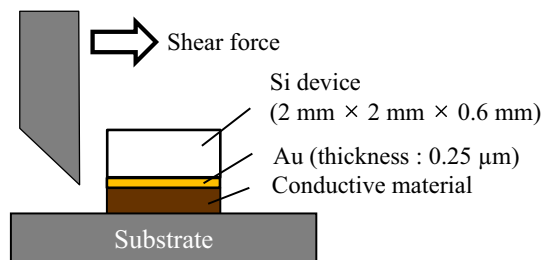**FIGURE 5** Structure of sample used for thermal conductivity measurements

FIGURE 6 Schematic of shear strength measurement of a conductive material

two requirements: minimization of organic residue and a deoxidation effect after the sintering process. At high temperatures, the durability and reliability of the TLPS conductive material is assured by minimizing the amount of organic residue. The interconnection mechanism shown in Figure 2B is achieved through solder wetting between Cu particles based on the deoxidation function of the volatile fluxing resin during the sintering process. As shown in Table 1, six volatile fluxing resins were mixed with Cu flakes and solder powders. The mixing weight percent ratio of the three components was 18:45:37. Figures 7 and 8 show the weight changes of the base resins (A, B, and C) and reductants (D and E) of the TLPS material, respectively, during the isothermal sintering process. The temperature reached 240 °C at a heating rate of 60 °C/min and was maintained at 240 °C for about 10 min. The evaporation of the volatile fluxing resins in the TLPS materials started slowly at about 100 °C during the heating process. The weight change of the volatile fluxing resins B/D, C/D, B/E, and C/E remained constant when the temperature reached 240 °C, whereas the weight change of A/D and A/E decreased under isothermal conditions at this temperature.

After the isothermal sintering process is applied, as shown in Figures 7 and 8, the residue weights of the volatile fluxing resin in the TLPS materials are summarized in Figure 9. The volatile fluxing resins B/D and C/E showed a weight percent of 3.7 and 1.8, respectively. As indicated in Table 2, the lowest and highest boiling points of the resin compositions A/E and B/D are 185 °C/200 °C and 242 °C/286 °C, respectively. However, the residue weight of the highest boiling point resin B/D was lower than that of the lowest boiling point resin A/E. Unexpectedly, the lowest residue weight of the TLPS material was found in resin C/D with a boiling point of 198 °C/286 °C, as indicated in Figure 9. Thus, the resin compositions B/D, C/D, and C/E in the present study can be used as candidate materials for a TLPS resin in terms of the residue weight.

The paste was printed and sintered, as shown in Figure 4, to measure the volume resistivity of the sintered TLPS materials. The sample shown in Figure 10 is a 40 mm × 40 mm wide TLPS paste printed on a 0.6-mm thick high-resistivity
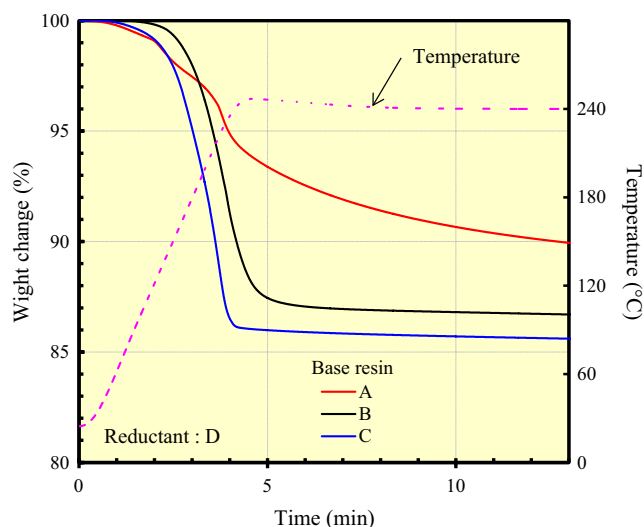

FIGURE 7 Weight change of TLPS material mixed with volatile fluxing resins (A/D, B/D, and C/D) and metal fillers
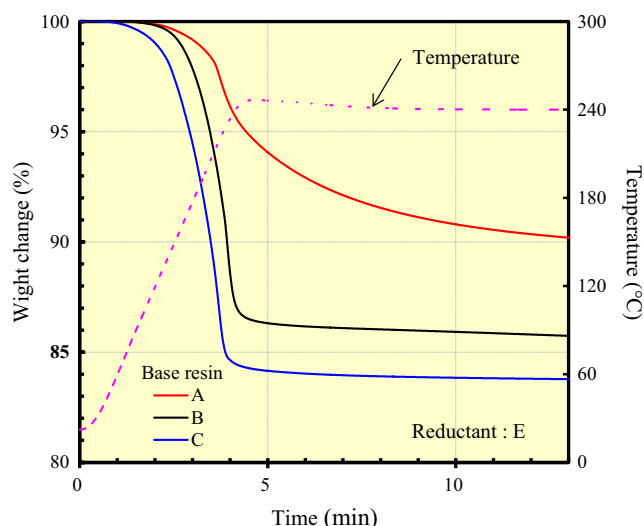

FIGURE 8 Weight change of TLPS material mixed with volatile fluxing resins (A/E, B/E, and C/E) and metal fillers

silicon substrate. The sintering process was continued for 10 min after reaching 240 °C, at a heating rate of 60 °C/min in a 1050 ppm oxygen containing environment. The average thickness of the sintered TLPS on the high-resistivity silicon substrate was about 25 μm. For both reductants (D and E), the volume resistivity of the TLPS using base resins A and B was lower than that of base resin C. In addition, the volume resistivity of TLPS using base resin C was higher than that of base resins A and B. The lowest volume resistivity of 81 μΩ-cm, as shown in Figure 11, was obtained for the TLPS with a volatile fluxing resin formulation of B/D, despite the resin formulation C/D showing the lowest residue weight, as indicated in Figure 9. Finally, the resin B/D was determined to have the optimum composition of the volatile
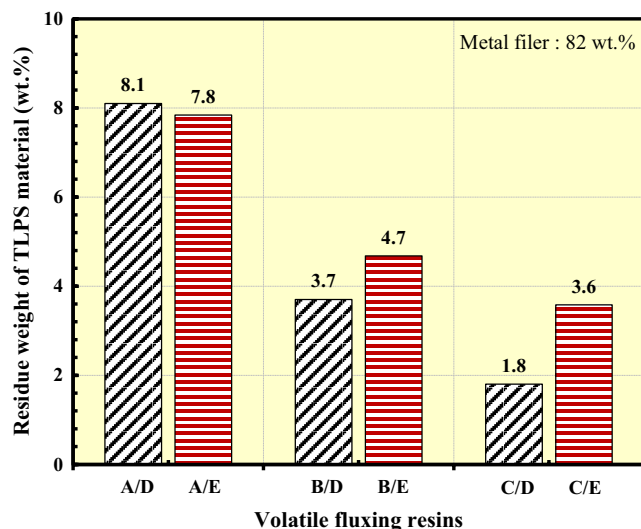
FIGURE 9 Residue weight of volatile fluxing resin in TLPS materials after isothermal sintering process
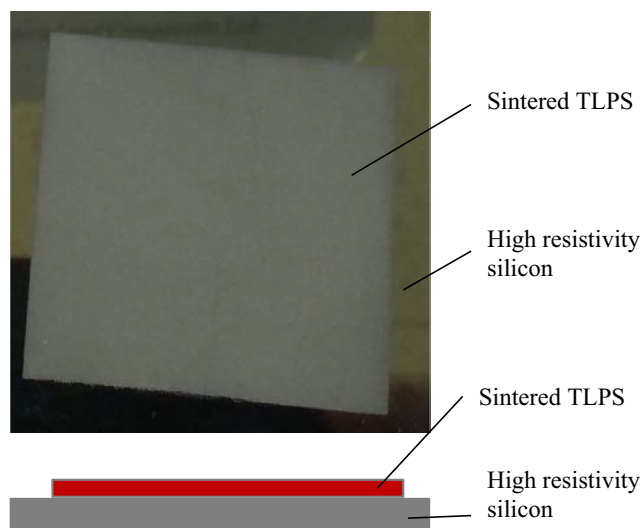

FIGURE 10 Photograph of sintered TLPS material on a high-resistivity silicon substrate

fluxing resin, taking into account the minimum residue of the resin and the lowest volume resistivity after the sintering process.

As shown in Figure 12, TLPS was performed at a processing temperature of 240 °C, and electrical and thermal conductive paths were also very well generated during the sintering process. From the SEM-EDX, the metal atomic composition of TLPS was observed as 6Ag/57Sn/37Cu. For characterization of the TLPS material including chemical and metallic components, dynamic DSC with a heating rate of 10 °C/min was performed, as shown in Figure 13. From Figures 13A and 12B, Ag-coated Cu does not show any exothermic or endothermic phenomena, whereas the SAC305 solder clearly shows sharp endothermic melting at 223 °C. Figure 13C shows the dynamic DSC result of a mixture of
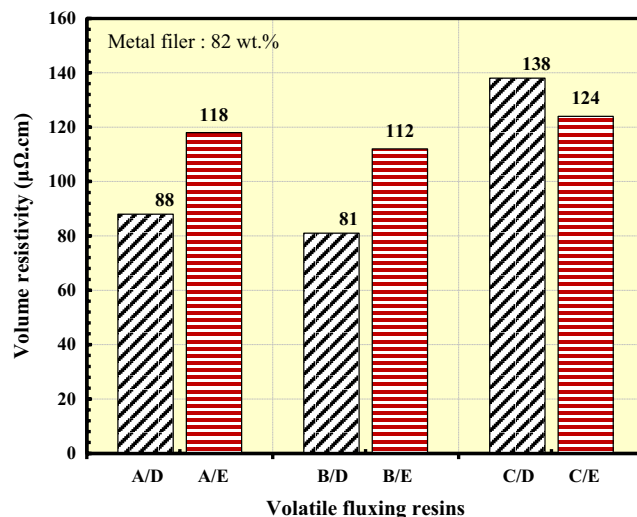

FIGURE 11 Measured volumetric resistivity of sintered TLPS materials on high-resistivity silicon substrates

Ag-coated Cu and SAC305 solder without any organic materials. As the temperature increases, as shown in Figure 13C, two melting points and one exothermic peak are observed. The first endothermic peak near 220 °C corresponds to the melting of the SAC305 solder and the second endothermic peak was assumed to be due to the intermetallic compound produced by the metallic reaction between the Ag-coated Cu and SAC305, because the exothermic peak was observed at near 230 °C after the melting point of the SAC305 solder. Thus, the exothermic peak is believed to have been caused by the partial metallic reaction from the physical contact between the molten SAC305 solder and Ag-coated Cu despite the incomplete removal of the solder oxide. Figure 13D shows the dynamic DSC result of the TLPS resin. Exothermic peaks indicating a chemical reaction were not found in the volatile fluxing resin, and one large endothermic
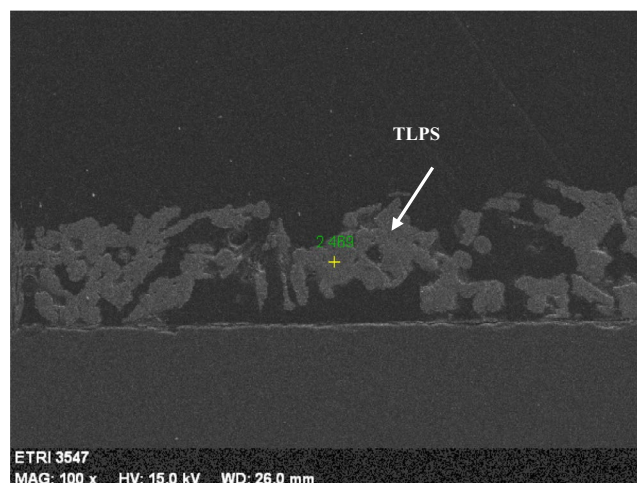

FIGURE 12 SEM-EDX picture of cross-sectional sintered TLPS material on a high-resistivity silicon substrate
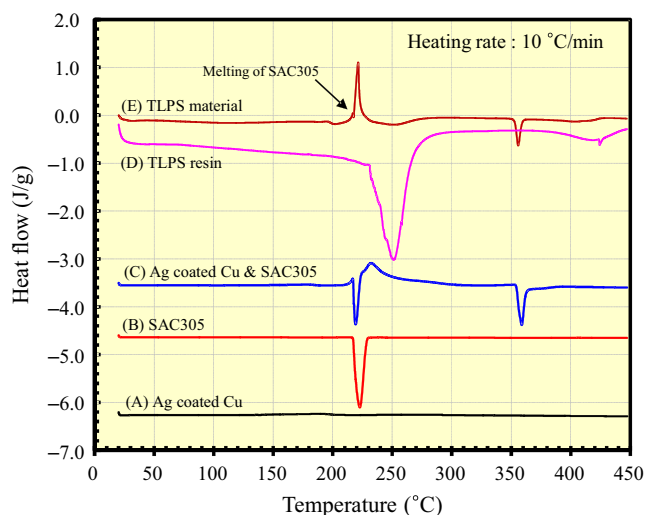
FIGURE 13 Dynamic DSC measurement results with heating rate of 10°C/min for TLPS material and its components

FIGURE 14 Comparison of dynamic DSC measurement results between the first and second runs of TLPS material with a heating rate of 10 °C/min

peak at about 250 °C, indicating the melting of the reductant in the TLPS resin, was observed.

As expected, no crosslinking reaction was observed in the TLPS resin. Figure 13E shows a dynamic DSC of a TLPS material composed of a volatile fluxing resin, Ag-coated Cu, and SAC305 solder. Two endothermic and one exothermic peaks were observed. Figure 13E shows a large exothermic peak at 221 °C immediately after the melting of SAC305 at 217 °C. This exothermic phenomenon was presumed to be caused by a metallic reaction between the deoxidized solder and Ag-coated Cu. The second melting peak was observed at 356 °C, as indicated in Figure 13E. Figure 14 shows a comparison of the first and second runs of a dynamic DSC using the TLPS material. In the second run of the dynamic DSC as shown in Figure 14B, only one endothermic peak was detected at 356 °C without the melting of SAC305 or any exothermic peak from the metallic reaction. This lack of an exothermic phenomenon indicates that the SAC305 solder completely reacted with the Ag-coated Cu, and the volatile fluxing resin was nearly completely evaporated during processing. As can be seen in Table 4, NIST [12] reported that a phase change of $Ag_3Sn + Cu_6Sn_5 \rightarrow$ liquid $+ Cu_3Sn$ occurs at 356.2 °C owing to the metallic reaction, which is consistent with the present research results. Therefore, it is believed that the melting peaks shown in Figure 14A and 14 are consistent with the phase change as shown in Table 4, as mentioned in [12].

Figure 15 shows the volume resistivity according to the metal filler amounts. The volume resistivity decreased linearly with the increase in filler content, which is thought to be due to the increased density during the processing as the filler amount increased. The viscosity of the TLPS material should be optimized for the screen printing process. The TLPS materials having filler amounts of 84 wt.% and 86 wt.% showed
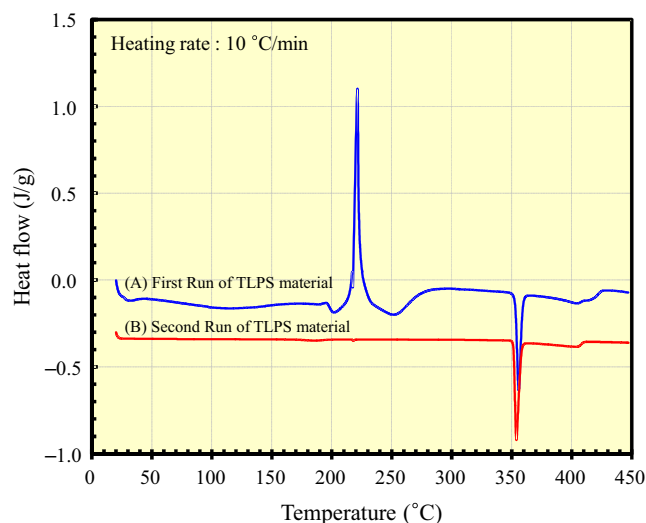
TABLE 4 Calculated invariant equilibria for intermetallic compound, from [12]

Reaction	Phase	Mass% Ag	Mass% Cu	Mass% Sn
$Ag_3Sn + Cu_6Sn_5 \rightarrow$ Liquid $+ Cu_3Sn$ (356.2°C)	Liquid	19.26	5.86	74.88
	Cu_3Sn	0	61.63	38.37
	Ag_3Sn	73.17	0	26.83
	Cu_6Sn_5	0	39.07	60.93

viscosities of 105 Pa.s and 243 Pa.s, respectively. The metal filler content of the TLPS material increased to only 2.0 wt.%, but the viscosity at 86 wt.% TLPS was 2.3 times higher than that at 84 wt.% TLPS.

To confirm the effects of the oxygen content in the SMT oven during the processing, screen-printed TLPS samples were processed at various oxygen concentrations and their volume resistivity was measured, as shown in Figure 16. As the oxygen concentration increased, the volume resistivity increased rapidly and reached 31 000 μΩ-cm under an air environment (oxygen 210 000 ppm). Therefore, it is necessary to control the oxygen content in the oven environment during processing to obtain a high-quality conductive material.

To increase the bonding strength, TLPS materials with a filler content of 84 wt.% were prepared by varying the amount of the adhesion promoter. Figure 17 shows samples for measuring the shear strength of a TLPS material processed on silicon and ceramic substrates. The measured shear strength of the processed TLPS material with different amounts of adhesion promoter is shown in Figure 18. On both substrates, the shear strength generally increased with an increase in the adhesion promoter for up to about 9 phr, and decreased in both cases after 9 phr. The shear
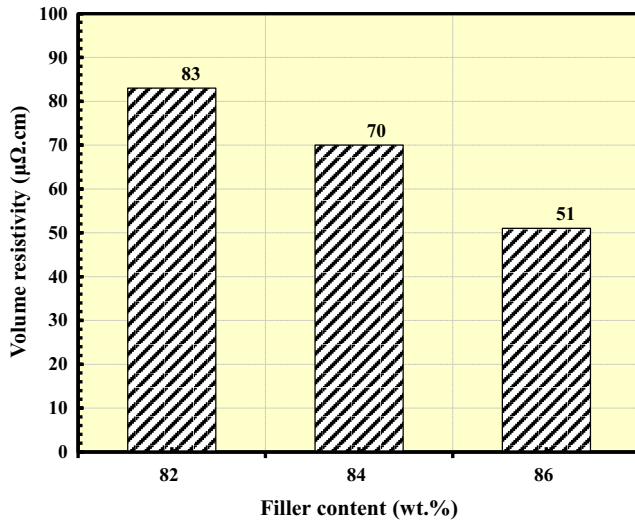
FIGURE 15 Measured volumetric resistivity of sintered TLPS materials on high-resistivity silicon substrates with different metal filler contents
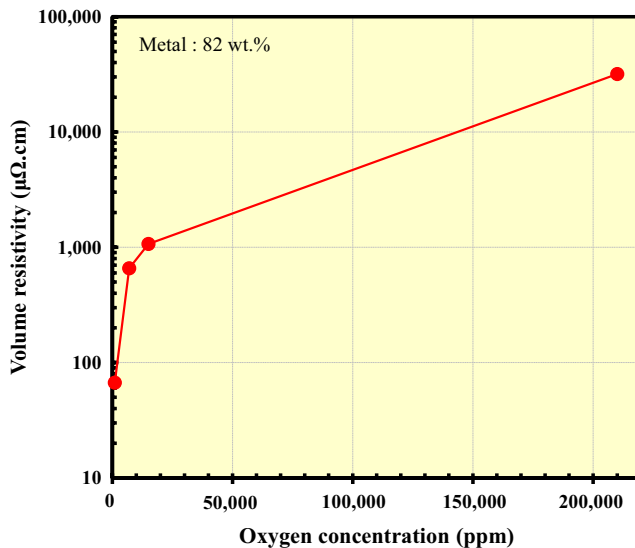

FIGURE 16 Measured volume resistivity of sintered TLPS materials on high-resistivity silicon substrates with different oxygen concentrations

FIGURE 17 Photographs of the sample to measure the shear strength of processed TLPS material on: (A) Silicon and (B) Ceramic substrates
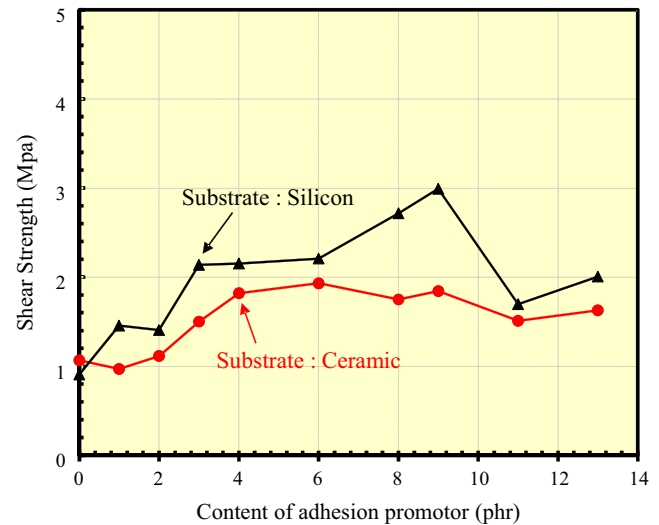
FIGURE 18 Measurement of shear strength of processed TLPS material with different amounts of adhesion promoter
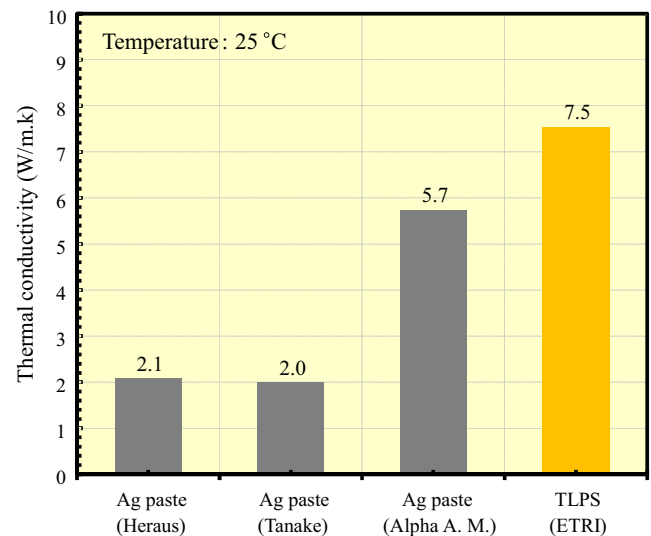
FIGURE 19 Comparison of measured thermal conductivity of TLPS material and commercialized Ag pastes

strength of the TLPS material with the silicon substrate generally appeared to be higher than that with the ceramic substrate. In the present research, an adhesion promoter of 4 phr was used in the composition of the TLPS material. Henkel reported that the adhesion strength of Ag sintering paste is higher than 20 MPa under 5–10 MPa bonding pressure on copper substrate with Au direct bonding [13]. For achieving high shear strength of a TLPS material, in a future study, the bonding process should be conducted under a high bonding pressure between the power device and the substrate.

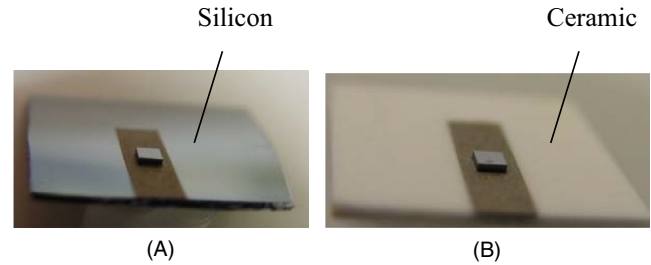A sample for measuring the thermal conductivity was prepared, as shown in Figure 5, for a comparison of the thermal conductivity of the TLPS material and various commercially available Ag pastes shown in Table 3. Figure 19 shows a comparison of the measured thermal conductivity of three

commercial Ag pastes and the TLPS material. The thermal conductivity of the TLPS material was higher than that of the other commercial Ag pastes. As described in Figure 5, all conductive pastes were coated on a Cu plate and cured according to the given curing process. The measured thermal conductivity is believed to include the interfacial effect between the conductive material and Cu plate, which is very similar to the actual application.

4 | CONCLUSION

A TLPS material including Ag-coated Cu, solder, and a volatile fluxing resin was proposed for a power device application and investigated as a conductive paste. The composition of the TLPS material was optimized in terms of the minimum residue of the fluxing resin, high volume resistivity, optimum viscosity for the screen printing process, and high shear strength. The formation of an intermetallic compound during the processing was also observed at 356 °C by using DSC. In particular, this TLPS material exhibited high reliability within a temperature range of up to 350 °C because the first melting of the TLPS material after processing was observed at 356 °C through dynamic DSC. From comparison with various commercially available Ag pastes, the TLPS material exhibited the highest thermal conductivity under the same sample preparation conditions. In conclusion, a low-cost TLPS material can be used for power device applications as a high-conductive adhesive instead of a high-cost Ag paste.

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ORCID

Yong-Sung Eom https://orcid.org/0000-0002-5678-9093

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AUTHOR BIOGRAPHIES

Yong-Sung Eom received his BS from the School of Aerospace and Mechanical Engineering at Korea Aerospace University, Goyang, Rep. of Korea and his MS from the Department of Aerospace Engineering at the Korea Advance Institute of Science and Technology, Seoul, Rep. of Korea, in 1988 and 1991, respectively. He worked at the Korea Institute of Aeronautical Technology (KIAT) at Korean Air Ltd., Incheon, Rep. of Korea as a design and process engineer for the composite materials of the MD-11 Aircraft Spoiler from 1991 to 1995. In 1999, he received his PhD from the Department of Material Science Engineering at École Polytechnique Fédérale de Lausanne, Lausanne, Switzerland. After returning to Korea, he worked at Hynix Semiconductor Ltd., Icheon, Rep. of Korea as a packaging engineer for memory devices from 2000 to 2001. Since 2001, he has been with Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, where he works as a packaging engineer. His research activities include the development of interconnection materials based on polymers for electronic packaging and process design for 3D-IC and MEMS packaging.

Keon-Soo Jang received his BS and MS in chemical engineering, focusing on polymers, from Sungkyunkwan University (Suwon, Rep. of Korea) in 2007 and 2009, respectively. He moved to the USA and was received his PhD in macromolecular science and engineering, focusing on multilayered coextrusion, robust supramolecular polymers and liquid crystal-embedded polymer composites, from Case Western Reserve University, Cleveland, OH, USA. After returning to Korea, he joined Samsung SDI-Lotte Advanced Materials in 2015 and has been involved in developing extrusion/injection-processed composites. Since 2016, he has been working at Electronics and Telecommunications Research Institute (Daejeon, Rep. of Korea), extending his interests to semiconductor packaging thermosetting materials. He is currently working as a faculty at the University of Suwon (Rep. of Korea).

Ji-Hye Son received her BS and MS from the Department of Advanced Organic Materials and Textile System Engineering at Chungnam National University, Daejeon, Rep. of Korea, in 2010 and 2012, respectively. Since 2012, she has been working for Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea. She has carried out research on the design and synthesis of organic polymers and the characterization of advanced polymers.

Hyun-Cheol Bae received his BS and MS in electrical engineering from Dongguk University, Seoul, Rep. of Korea, in 1999 and 2001, respectively. He joined the SiGe research team of Electronics and Telecommunications Research Institute (ETRI), Daejeon, Rep. of Korea, in 2001 and worked as a design and process engineer for MMIC and passive devices. He joined the packaging research group of ETRI in 2007 and has been working as a packaging engineer. In 2009, he received his PhD in electrical engineering from Chungnam National University, Daejeon, Rep. of Korea. His research interests include the design and fabrication of integrated passive devices, 3D stacked chip packaging using TSV, and wafer-level packaging for MEMS devices.

Kwang-Seong Choi received his BS degree from Hanyang University, Seoul, Rep. of Korea, and his MS and PhD degrees from Korea Advanced Institute of Science and Technology, Daejeon, Rep. of Korea. From 1995 to 2001, he was with Hynix Semiconductor Ltd., Icheon, Rep. of Korea, where he was involved in the development of chip scale packages, package-on-packages, and high-speed electronic packages for DDR, Rambus, and RF devices. Since 2001, he has been with Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, where he is a Principal Member of Engineering Staff. His research interests include the development of packaging materials and processes for 3D ICs with through silicon via (TSV), next-generation displays, and large array of thin devices.