Fast and Reliable Ag–Sn Transient Liquid Phase Bonding by Combining Rapid Heating with Low-Power Ultrasound

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Ag–Sn transient liquid phase (TLP) bonding is capable of producing joints with excellent mechanical and thermal properties, and with operation temperatures at or even above the processing temperature. However, reduction of the comparatively long processing times required to achieve high-quality joints is highly desirable. To this end, the use of fast heating and low-power ultrasonic (US) pulses for accelerated Ag–Sn TLP bonding is investigated with particular focus on defect generation and avoidance. Employing rapid heating with 20 Ks⁻¹, the process time can indeed be strongly reduced, yielding a high average shear strength of (58 ± 22) MPa. The large variance of the shear strength is caused by gas entrapment and pronounced lateral squeeze-out of liquid Sn, facilitated by the suppression of Ag₃Sn formation upon rapid heating. Introduction of a weak US pulse leads to enhanced Ag₃Sn formation and efficient removal of entrapped gas, and thus to avoidance of large-scale bond defects, which results in an enhanced shear strength of (104 ± 8) MPa.

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I. INTRODUCTION

JOINING for microelectronics still heavily relies on classical Sn-based solder systems, e.g., lead-free Sn–Ag–Cu solders. However, there is an increasing demand to manufacture joints with higher service temperatures (often in excess of 300°C), improved temperature stability, and better heat dissipation. Typical examples are die attach and heat-sink bonding for high-power modules and wide-bandgap semiconductor devices, but also the assembly of electronic devices for high-temperature environments. Although some (lead-free) solder systems with operation temperatures higher than Sn-based solders exist (e.g., Au–Si or Au–Ge), the likewise higher temperatures required for the joining process often bar their actual implementation, for instance due to temperature-sensitive components in the joining assembly, or due to joining equipment limited to lower process temperatures (e.g., standard reflow ovens or die bonders).

Transient liquid phase (TLP) bonding, or solid–liquid interdiffusion (SLID) bonding, is a dedicated joining technology that can fulfill the opposing demands of low process but high operation temperature: for TLP bonding, a metal interlayer (usually a thin metal foil or coating) with low melting point is sandwiched between two joining partners with a suitable metallic surface or surface metallization. Upon heating, the interlayer metal melts and reacts with the adjacent surface layers. The reaction between the different metals generally leads to the formation of one or more intermetallic compound (IMC) phases with high melting point. Consequently, after completion of the reaction, the re-melting temperature of the produced joint, and thus also the operation temperature, can be much higher than the process temperature.

Cu–Sn and Ag–Sn are two TLP systems that are principally well suited for electronics applications: both form IMC phases with high melting temperatures, and require only moderate process temperatures (typically between 250°C and 300°C). Both systems are well investigated with respect to their phase formation sequence and kinetics upon TLP bonding (e.g.,...
References 6–10). The use of Cu–Sn is particularly well documented for bonding processes on the wafer level, e.g., for realization of wafer-level interconnects\cite{11,12} as a replacement for Sn-solder bumping or for wafer-level packaging\cite{13–15}. However, Cu–Sn shows a pronounced tendency for Kirkendall voiding upon the formation of different Cu–Sn IMC phases, and for bond embrittlement, which tend to increase with an increasing amount of IMCs. Cu–Sn TLP processes are therefore better suited for wafer-level processes, for which bonding with very thin interlayers (≤ 4 μm), and thus very thin IMC product layers, can be readily implemented. In this regard, the system Ag–Sn offers some advantage: it features only two known IMC phases, Ag₃Sn (α) and ζ, and shows little tendency for Kirkendall voiding upon IMC transformation (Ag₃Sn → ζ). This leads to a comparably stable microstructure already upon formation of the Ag₃Sn phase during TLP bonding, but also upon subsequent formation of the ζ phase (e.g., upon prolonged aging at elevated temperatures)\cite{16,17,18}.

Accordingly, high-quality joints can be manufactured by Ag–Sn TLP bonding, offering high strength and high-temperature stability even for thicker joints. For instance, shear strengths up to 50 to 60 MPa are reported for TLP processes using Ag surface metallization and Sn foils of 8 to 10 μm thickness as interlayers and applying bonding pressures of ≤ 0.5 MPa\cite{18,19}. These mechanical values are considerably higher than for most Sn-based solders\cite{20}, and comparable to high-strength Ag-sinter joints\cite{21}. A further increase in shear strength to more than 100 MPa could be achieved by our group by replacing Sn foils with sputter-deposited Sn coatings\cite{17}. This comparatively high mechanical strength was directly achieved after completion of the TLP bonding process by Ag₃Sn formation, and was maintained after prolonged annealing upon Ag₃Sn → ζ transformation.

A central drawback of the reported Ag–Sn TLP processes are the process parameters required to obtain these high-quality joints: annealing for ≥ 60 minutes at 300 °C is reported, necessary to achieve complete transformation of Sn into Ag₃Sn and a low porosity within the joining zone\cite{18,17,19}. In addition, comparatively low heating rates of ≤ 15 K min⁻¹ were applied, as e.g., achievable with resistance-heating furnaces, which further increases the duration of the process. A reduction of the comparatively long processing times and high processing temperatures is highly desirable to reduce costs and the thermal load of the joining components during the process.\cite{22} Moreover, bonding equipment as used for die attach in microelectronics (e.g., die bonder, or heating stages) is often designed for fast heating rates (several Ks⁻¹ instead of several Kmin⁻¹), short processing times (s to min), and temperatures below 300 °C, and therefore not compatible with classical Ag–Sn TLP bonding processes.

Reduction of the process temperature, i.e., the isothermal annealing temperature, generally entails slower reaction kinetics due to lower thermal activation, and thus potentially leads to longer bonding times. For TLP bonding, it was also shown that the annealing temperature strongly influences the evolution of the microstructure, as well as the quality of the resulting bond\cite{18,19}. Careful adjustment of the annealing temperature is therefore necessary to achieve an optimum of bond quality and process time.

Shorter process times for TLP bonding may principally be realized straightforwardly by use of (ultra-)fast heating rates, as available on dedicated die bonder or heating stages. However, the question how very fast heating rates of several Ks⁻¹ affect the microstructural evolution during the TLP process, as well as the quality of the resulting bond, is not yet fully clarified\cite{23,24}.

Other approaches to accelerate the TLP process are for instance the use of (i) very thin interlayers to reduce the amount of material to be transformed (e.g., Reference 12), or of (ii) multi-layered foils\cite{24–26} and (iii) of powder mixtures\cite{27,28} to reduce the effective diffusion distances. However, these approaches typically require vacuum bonding systems and high bonding pressures\cite{12,24–26} or substantial amounts of flux to avoid oxidation of the powders, the latter resulting in a comparatively low bond quality\cite{27,28}.

A recently proposed approach to drastically reduce the process time for Ag–Sn and Cu–Sn TLP bonding is the use of ultrasound (US) upon bonding. US application during the liquid stage was shown to drastically shorten the isothermal holding time required to form a

| Case | Tₙ (°C) | Φ (Ks⁻¹) | tₐ (s) | t₁ (s) | tₐm (s) | tₐe (s) | tUS (s) | P_US (mWmm⁻²) |
|------|---------|----------|--------|--------|----------|----------|--------|-------------|
| 1    | 240     | 0.25     | 10     | 828    | 44       | 92       | —      | —           |
| 2    | 240     | 20       | 10     | 38     | 12       | 15       | —      | —           |
| 3    | 300     | 0.25     | 10     | 1080   | 296      | 344      | —      | —           |
| 4    | 300     | 20       | 10     | 53     | 27       | 30       | —      | —           |
| 5    | 240     | 20       | 180    | 208    | 182      | 185      | —      | —           |
| 6    | 300     | 20       | 180    | 223    | 197      | 200      | —      | —           |
| 7    | 240     | 20       | 180    | 208    | 182      | 185      | 1      | 62.5        |
| 8    | 240     | 20       | 180    | 208    | 182      | 185      | 1      | 62.5        |

| Table I. Time–Temperature Profile Parameters |

\[ Tₙ; \text{isothermal annealing temperature; } Φ; \text{heating rate; } tₐ; \text{isothermal holding time; } t₁; \text{total process duration (between heating from 40 °C and cooling to 150 °C with } -5 \text{ Ks}^{-1}); tₐm; \text{liquids time above the melting temperature of pure Sn } (T_{m,Sn} = 231.93 °C); tₐe; \text{liquids time above the eutectic temperature of Ag–Sn } (Tₙ = 220.5 °C); tUS; \text{duration of the US pulse; } P_US; \text{power density of the US pulse.} \]
fully reacted TLP bond down to a few seconds.\textsuperscript{[39–33]} For instance, for Ag–Sn TLP complete Sn consumption could be achieved upon annealing for 15 seconds at 280 °C with simultaneous application of US.\textsuperscript{[31]} yielding joints with an average shear strength comparable to standard Ag–Sn TLP processes (see above). Typical US powers reported for US-assisted TLP bonding range between 250 and 750 W, corresponding to power densities of \textit{ca.} 9 W mm\textsuperscript{-2}\textsuperscript{[30,31]} up to several hundreds of W mm\textsuperscript{-2} (in the case of very high power densities, due to the high energy input no external heating is required to liquefy the Sn interlayer\textsuperscript{[38]}). Although US-supported TLP bonding allows to reduce the process times drastically, utilization of such high power densities requires dedicated equipment for US bonding, and can be problematic in case of mechanically sensitive devices.

In the present work, the use of fast heating combined with low-power US agitation for accelerated foil-based Ag–Sn TLP bonding is explored. To this end, the effect of rapid heating rates on the bonding process is investigated with particular focus on the formation of the Ag\textsubscript{3}Sn phase and the generation of bonding defects upon rapid heating and subsequent isothermal annealing at different isothermal temperatures. In a second step, the introduction of weak US pulses upon TLP is studied. On this basis, an optimized bonding process with reduced process time and temperature is proposed, which combines the advantages of rapid heating and US application to realize high-strength Ag–Sn TLP bonds.

## II. MATERIALS AND METHODS

For the TLP bonding experiments, oxygen-free high thermal conductivity (OFHC) copper plates of 1 mm thickness were electrolytically coated with 15 μm of pure Ag (Collini, Switzerland; see Figure 1 for a schematic of the joining setup). The plates were subsequently cut into smaller plates of 4 × 4 mm\textsuperscript{2} (upper substrate) and 10 × 20 mm\textsuperscript{2} (lower substrate) with a diamond-wire saw, and afterward deburred. The surface roughness and the flatness of the cut substrates were determined using a Bruker DektakXT stylus profilometer. The resulting average surface roughness of the electroplated surface amounts to \( R_a = 0.07 \) μm. The flatness of the specimens was found to be about 1 μm, measured over a distance of 3 mm for the 4 × 4 mm\textsuperscript{2} substrates. As interlayers, Sn foils of 8 μm thickness and purity of 99.75 pct (Goodfellow, UK) were used. To achieve a complete TLP reaction of 8 μm Sn into Ag\textsubscript{3}Sn, a (combined) layer thickness of about 15 μm Ag would be sufficient. In the present case, an excess amount of Ag (2 × 15 μm) was deliberately chosen to allow for a potential further transformation of Ag\textsubscript{3}Sn into Ag-rich \( \zeta \) phase. To reduce the amount of surface oxides, the Ag-plated substrates were etched in 20 wt pct HNO\textsubscript{3}, and the Sn foils were etched in 10 wt pct HCl immediately before bonding, rinsed twice in demineralized water, and kept in isopropanol until assembly.

The bonding experiments were executed on a manual flip-chip bonder (Fineplacer Pico Ma, Finetech) equipped with a heating stage and chip-heating tool for fast heating with a maximum rate of 20 K s\textsuperscript{-1}. A high parallelism between upper and lower substrate proved to be crucial to achieve a high bonding quality. The parallelism between heating stage and chip-heating module was therefore adjusted with an error less than 1/500 deg. For the bonding experiments, a constant pressure of 6 N/16 mm\textsuperscript{2} = 0.375 MPa (force-controlled) was used. Upon bonding, the specimens were flushed with Ar (4.8 N) in order to suppress oxidation, especially in the case of longer bonding times.

For the US-supported bonding experiments, the chip-heating tool was replaced by an unheated US tool originally designed for so-called thermosonic bonding (cf. Figure 1(c)). Thermosonic bonding is a combination of friction bonding by US agitation and thermal bonding without involvement of a liquid phase,\textsuperscript{[37]} and is a well-established technology for flip-chip die attach \textit{via} metal studs. In order to avoid damaging of the semiconductor chip, comparatively low US powers are usually applied.\textsuperscript{[38]} Accordingly, US tools for thermosonic flip-chip bonding are typically limited to low US powers, here 40 W. The US tool, operating at 40 kHz, causes a one-directional flexural motion of the tool tip with a lateral amplitude of \textit{ca.} 2 to 4 μm (the vertical component is negligible). Single-sided heating with only the bottom heating stage causes a temperature gradient across the bonding setup during TLP bonding, \textit{i.e.}, also across the specimen. The resulting temperature shift amounted to 5 K, quantified by monitoring the melting point of the pure Sn interlayer \textit{via} a separate process camera, and could be corrected by increasing the temperature of the bottom heating stage.

The effects of different temperature profiles and of US application on the TLP bonding process were assessed \textit{via} microstructural investigations by scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS), as well as by mechanical testing. For the investigation of the microstructure after bonding, cross-sections were prepared by wire cutting followed by ion milling (Hitachi IM4000). The cross-sections were analyzed with SEM–EDS using a Hitachi S3700N equipped with an Octane Pro EDS detector (EDAX/Ametek). Imaging of different phases by SEM is preferably done in back-scatter electron (BSE) mode. However, due to the similar atomic masses of Ag (atomic number 47) and Sn (atomic number 50), the various phases in the Ag–Sn system (\textit{i.e.}, Ag, Sn, Ag\textsubscript{3}Sn and \( \zeta \)) have a similar Z-contrast (mass contrast). This leads to only weak contrast variations between the phases in SEM–BSE. Phase identification was therefore corroborated by additional EDS spot measurements. To quantify the mechanical stability, shear tests were performed using a Walter & Bai shear tester. The upper substrate was sheared off with a tungsten carbide shear blade at a distance of 100 μm from the joining zone with a constant shear rate of 0.6 mm min\textsuperscript{-1}. 
III. RESULTS

A. Microstructural Evolution upon Slow and Fast Heating

In a first step, the evolution of the microstructure upon heating was investigated for a conventional Ag–Sn TLP bonding process, i.e., without the application of US. Two representative heating rates were considered: a conventional heating rate of 15 K min\(^{-1}\) = 0.25 K s\(^{-1}\) up to an annealing temperature of 240°C (case 1 in Table I and Figure I(b)), and a high heating rate of 20 K s\(^{-1}\) (case 2). In both cases, heating was followed by a short holding step of 10 seconds at 240°C for temperature equilibration (for fast heating, melting of the Sn foil was observed after 3 seconds into the holding step), and subsequent rapid cooling with 5 K s\(^{-1}\) = 300 K min\(^{-1}\) down to 150°C. The annealing temperature \(T_a\) = 240°C is slightly above the melting point of pure Sn at \(T_m,Sn\) = 231.93°C, and about 20 K above the eutectic temperature of Ag–Sn at \(T_e\) = 220.5°C (Figure 1(a)). Application of the respective heating rates leads to pronouncedly different total process times \(t_a\), as well as liquidus times \(t_l,Sn\) and \(t_e\) above \(T_m,Sn\) and \(T_e\), respectively (Table I; Figure 1).

Slow heating to 240°C (case 1) resulted in the formation of localized clusters of Ag\(_3\)Sn IMC grains along the original Ag surfaces (see Figure 2 and Table II). These clusters comprise small, scallop-type particles with diameters of about 0.5 to 1.5 μm, but also some large and pronouncedly faceted particles with diameters up to 5 μm almost crossing the entire bonding zone. Next to the particle clusters, the bonding zone still consists of unreacted Sn, re-solidified upon cooling. According to SEM–EDS (Table II), the re-solidified Sn phase in the vicinity of the Ag\(_3\)Sn-particles already contains several percent of Ag (likely in the form of extremely fine Ag\(_3\)Sn precipitates). This indicates that locally a near-eutectic mixture with a solidus temperature of \(T_s\) = 220.5°C was established. At some places within the bonding zone (Figure 2, case 1-overview), a number of extended pores can be observed, resulting from air entrapped between the Sn foil and the metallized substrates. The pores are not yet fully spheroidized, hinting at a still sluggish behavior of the liquid Sn phase just above the melting point. Application of a fast heating rate of 20 K s\(^{-1}\) up to 240°C (case 2 in Table I and Figures 1 and 2) leads to a similarly inhomogeneous bond microstructure: A small number of Ag\(_3\)Sn grains are already formed at some locations along the original Ag surfaces. However, in contrast to case 1, cluster formation is far less pronounced. Moreover, the individual Ag\(_3\)Sn grains are of much smaller size, and do not yet protrude far into the unreacted Sn. As in the previous case, entrapped air produces extended pores which are not yet spheroidized.

In a second step, the microstructural evolution upon heating to 300°C was investigated, which corresponds to the optimum annealing temperature reported for furnace-based Ag–Sn TLP processes\([10,17]\). Heating to 300°C with a low heating rate of 0.25 K s\(^{-1}\) (case 3 in Table I) resulted in the formation of two densely packed bands of Ag\(_3\)Sn IMC grains with particle diameters of about 2 to 4 μm, but also of a few massive Ag\(_3\)Sn grains stretching across the entire bonding zone (case 3 in Figure 2; cf. case 1). Between the IMC grains, large bands of Ag\(_3\)Sn IMC grains with particle diameters of about 2 to 4 μm, but also of a few massive Ag\(_3\)Sn grains stretching across the entire bonding zone (case 3 in Figure 2; cf. case 1). Between the IMC grains, large bands of Ag\(_3\)Sn IMC grains with particle diameters of about 2 to 4 μm, but also of a few massive Ag\(_3\)Sn grains stretching across the entire bonding zone (case 3 in Figure 2; cf. case 1). Between the IMC grains, large bands of Ag\(_3\)Sn IMC grains with particle diameters of about 2 to 4 μm, but also of a few massive Ag\(_3\)Sn grains stretching across the entire bonding zone (case 3 in Figure 2; cf. case 1). Between the IMC grains, large...
240 °C

20 K/s
10 s

2µm
10µm

case 1

200 °C

20 K/s
180 s

2µm
10µm

case 5
pockets of residual Sn exist. In addition to the Sn pockets, a small number of large spheroidized pores can be observed in the center of the bonding zone, as well as a few small spherical pores between the already formed Ag₃Sn grains.

In case of the specimen heated with a high heating rate of 20 Ks⁻¹ to 300 °C (case 4 in Table I and Figure 2), two densely packed bands of Ag₃Sn grains are formed along the original Ag surfaces, as well. The size of the Ag₃Sn grains is slightly smaller than in case 3, with grain diameters of about 1 to 4 μm. Some individual grains again extend across the entire bonding zone, or impinge onto each other from both sides of the bonding zone. Notably, the average size of the Sn pockets is much smaller than in the case of the slowly heated specimen (case 3). This coincides with a generally lower thickness of the bonding zone than observed for the specimen (case 3). The reduction of the bond thickness can be attributed to lateral outflow of Sn observed upon heating to 300 °C with 20 Ks⁻¹. In clear difference to the previous cases, extended gaps, respectively, pore bands, are now present at many locations in the center of the bonding zone.

B. Microstructural Evolution upon Isothermal Holding: Mechanical Testing

To assess the time necessary for completing the TLP reaction after rapid heating to 240 °C and to 300 °C, the microstructural evolution upon isothermal annealing was studied. After annealing for 180 seconds at 240 °C (case 5 in Table I and Figure 2), the bonding zone features small Ag₃Sn grains with about 2 to 3 μm mixed with a few pores of similar size, and a number of very small spherical pores. No residual Sn phase was found for the present conditions. Notably, similar to case 3 (300 °C, 20 Ks⁻¹), extended pore bands have been formed between the well-bonded regions. In addition, the thickness of the bonding zone is strongly reduced, accompanied by a substantial lateral outflow of Sn (the absolute amount of outflowing Sn is still comparatively small due to the low thickness of the Sn foil).

At 300 °C, annealing for 180 seconds (case 6 in Table I and Figure 2) primarily leads to continued growth of the large Ag₃Sn grains that were already present at the beginning of the isothermal annealing step (cf. case 4). However, in contrast to annealing at lower temperature, some pockets of residual Sn are still present between the Ag₃Sn grains after 180 seconds. Only after prolonged annealing for 10 minutes at this temperature, complete transformation of the residual Sn was reached. The prolonged annealing had no effect on the gaps already present within the bonding zone at the beginning of the isothermal step, i.e., no lateral or vertical shrinkage of the gaps was observed. For the present conditions, no indications for the transformation of Ag₃Sn into Ag-rich ζ phase were found yet (cf. Reference 17).

To quantify the mechanical stability of the fully transformed specimens, shear tests were performed for joints annealed for 180 seconds at 240 °C, resulting in an average shear strength of (58 ± 22) MPa (Figure 3). Similar values were obtained for joints annealed for 10 minutes at 300 °C. According to SEM–EDS analysis of the fracture surfaces, fracture occurred in a brittle way within the Ag₃Sn by intercrystalline (or transcrystalline) fracture with negligible plastic deformation (also cf. the case with US application below). This means that the cohesion between the residual Ag and the grains of the newly formed Ag₃Sn layer is higher than between the Ag₃Sn grains. Notably, the measured shear strength values showed a very large scatter with a measured minimum value of 41 MPa and a maximum value of 105 MPa, as represented by the box plot in Figure 3 and the large variance of the average value. The measured shear strength strongly correlated with the amount of bonding defects visible in the fracture surfaces after mechanical testing: a lower amount of unbounded areas, corresponding to the pore bands visible in the cross-sectional analysis (cf. Figure 2), resulted in a higher shear strength, and vice versa. For the present joining setup, the rate of cooling had no effect on the measured shear strength.

C. US-Supported TLP

Based on the optimized time–temperature profile with rapid heating to 240 °C, the use of additional (lateral) US agitation during the TLP process was tested for its potential to further reduce the bonding time and improve the quality of the bonds. Compared to previous works, the US pulses were deliberately limited to low powers and short durations to minimize the amount of mechanical stress exerted on the components.

Application of US pulses proved to be most effective if placed shortly after the melting of the Sn foil. To study the effect of the weak US pulses on the microstructural evolution and bond quality, different US powers and durations were applied starting from an instrumentally defined minimum of 0.3 W for 0.2 seconds. Case 7 in Figure 4 shows the bond microstructure obtained for rapid heating with 20 Ks⁻¹ to 240 °C as in case 2 (see above), but with an additional US pulse of 1 W, corresponding to 1/16 Wmm⁻² = 62.5 mWmm⁻², for 1 second during the holding step (cf. Table I): compared to the specimen without use of US (case 2 in Figure 2), a much more pronounced formation of small Ag₃Sn grains with small pockets of residual Sn is observed. In addition, the amount of large gaps is strongly reduced, leading to a considerably more uniform bond microstructure. Analogous to the TLP process without application of US, massive lateral outflow of liquid Sn was observed, leading to a similar final bond thickness. Complete transformation into Ag₃Sn was again achieved after annealing for 180 seconds (case 8 in Figure 4).
A comparatively weak pulse of 62.5 mW mm$^{-2}$ for 1 second already sufficed to considerably improve the quality of the bond: after US application and immediately quenching, shear strengths of about 80 MPa were obtained, whereas application of longer US pulses lead to a decrease of shear strength. After annealing for 180 seconds to complete the Ag$_3$Sn formation, an average shear strength of (104 ± 8) MPa was finally reached (Figure 3). In contrast to bonding without US application, the deformation curves upon shear testing showed pronounced plastic behavior before bond failure, caused by the plastic deformation of the OFHC Cu substrates. Analysis of the fracture surfaces indicated again brittle intercrystalline (or transcrystalline) fracture within the Ag$_3$Sn layer (Figure 5). Shear marks indicating plastic deformation were observed only at the immediate edge of the substrate face in contact with the shear blade, where also the plastic deformation of the substrate was the highest (region 1 in Figure 5(b)). These were followed by a region of brittle trans- or intercrystalline fracture within the Ag$_3$Sn layer (region 2). This is most likely the region where the failure of the bond was initiated (localization of the failure origin was not possible due to the occurrence of brittle fracture, which shows little to no discernible features of crack propagation). Next to this region, large areas with additional shear marks could be observed (region 3). These are most likely post-failure shear marks created by the moving shear blade and/or upper substrate upon failure of the bond.

**Table II. Results of EDS Spot Measurements at the Positions Marked in Figures 2 and 4**

| Position | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|----------|---|---|---|---|---|---|---|---|---|
| Ag/at. pct | 5.8 | 74.2 | 73.4 | 100.0 | 3.8 | 72.3 | 0.0 | 1.8 | 75.7 |
| Sn/at. pct  | 94.2 | 25.8 | 26.6 | 0.0 | 96.2 | 27.7 | 100.0 | 98.2 | 24.3 |

The measurements indicate the transient appearance of a liquid Sn phase with some dissolved Ag, and the formation of Ag$_3$Sn. The Ag-rich phase was not observed.

![Figure 3](image1.png)

**Fig. 3**—Joint shear strength obtained for rapid heating with 20 K/s to 240°C and isothermal holding for 180 seconds, and for additional application of an US pulse with 1 W for 1 second at the beginning of the isothermal step. The data are represented as Tukey-type box plots. Red asterisk: average value; red bar: median value; blue bars: lower (25 pct) and upper (75 pct) quartile; whiskers: maximum 1.5 inter-quartile range; red cross: outlier (Color figure online).

![Figure 4](image2.png)

**Fig. 4**—Back-scatter electron images of joint microstructures obtained with additional application of a short US pulse at the beginning of the isothermal step (cases 7 and 8 in Table I). A: Sn phase, B: Ag$_3$Sn, C: Ag phase, D: different types of porosity. The border between Ag$_3$Sn (B) and Ag (C) is indicated by dashed lines on the left side of the micrographs. The numbers mark the positions of supplementary EDS measurements (see Table II).
IV. DISCUSSION

A. Ag₃Sn Formation upon Heating and Isothermal Holding

Analysis of the microstructural evolution upon heating revealed a pronounced influence of the heating rate on the nucleation and growth of the Ag₃Sn phase during the heating step (also see schematic in Figure 6). Upon heating to 240°C, i.e., just above the melting point of pure Sn, slow heating with 0.25 Ks⁻¹ (case 1) and fast heating with 20 Ks⁻¹ (case 2) both lead to formation of more or less isolated Ag₃Sn clusters, but with largely different sizes of the Ag₃Sn grains. The formation of isolated IMC clusters can be explained by the usage of
Sn foils as interlayer material: due to the finite flatness of the interlayer foil in combination with the comparatively low bonding pressure, immediate contact between the Sn foil and the Ag metallizations is only realized in a few points along the bonding interface. Hence, solid-state interdiffusion upon heating is only possible at a few isolated locations. Due to the very limited solubility of Ag in solid Sn, even minute diffusion of Ag into Sn upon heating can lead to a drastic decrease of the liquidus temperature of the (Sn) solid solution down to the eutectic temperature of the (Sn) + Ag,Sn equilibrium (see Figure 1(a)). Hence, local melting can occur once the eutectic temperature of 220.5°C is surpassed upon heating. The presence of a liquid phase can then accelerate the local erosion of the Ag metallization into the now liquefied Sn, and consequently also dramatically accelerate the formation of Ag,Sn via precipitation from the supersaturated liquid phase. The much larger particle size in the case of slow heating (case 1) than in the case of fast heating (case 2) can be directly attributed to the much longer timespan available for interdiffusion, dissolution of Ag, and for Ag,Sn grain growth (Table 1). A pronounced influence of the heating rate on the IMC formation was also reported in Reference 23 for Cu–Sn TLP. The anisotropic shape exhibited by the particularly large Ag,Sn grains is likely caused by pronounced preferential growth of these grains over a long period upon slow heating.

Direct heating to 300°C leads to substantial formation of Ag,Sn grains for both heating rates (cases 3 and 4). This indicates that, once the Sn foil is completely melted, rapid nucleation and (continued) growth of Ag,Sn sets in at every location of the bonding zone. In both cases, a pronounced scallop-type growth morphology of the Ag,Sn particles is found, i.e., more or less globular particles are formed extending from the original Sn–Ag interfaces into the bonding zone. This morphology is typical for IMC formation at a solid–liquid boundary, where rapid growth of the IMC scallops can occur by dissolution of the solid phase via liquid channels between the individual IMC grains, and precipitation of the IMC phase from the liquid phase. The reduced scallop size observed in the case of rapid heating can once again be attributed to the much shorter liquidus time (possibly in combination with a reduced availability of Sn due to the lateral outflow, see below). Notably, compared to the results from a conventional foil-based Ag–Sn TLP furnace process with the same nominal heating rate of 0.25 Ks⁻¹ the TLP reaction in the present case has proceeded much further, and much less residual Sn is observed. This difference may be attributed to a better surface oxide removal, better contact between foil and substrates upon heating, and/or a more efficient heat transfer due to the direct contact of the specimen with the heating stage and the heated placement tool in the present case. After rapid heating to the respective annealing temperature, isothermal holding at 240°C (case 5) or 300°C (case 6) produced joint microstructures with pronouncedly different degree of refinement: Annealing at the higher temperature of 300°C resulted in a smaller number of considerably larger Ag,Sn IMC grains than at 240°C, thus strictly following the predictions of classical nucleation and (thermally activated) growth theories.[42]

Notably, annealing at 300°C required considerably more time for completion of the TLP reaction than in the case of 240°C. This observation may, at first glance, seem counter-intuitive with respect to faster growth of the IMC phase expected at higher temperature due to faster solid-state interdiffusion of Ag and Sn, as well as faster dissolution and diffusion of Ag in liquid Sn. This observation can be explained by the microstructural refinement at the lower annealing temperature: After rapid initial growth of the IMC particles, in later stages of the TLP reaction, the scallop-type IMC growth results in the formation of isolated pockets of residual Sn that are fully surrounded by Ag,Sn grains. Further reaction of Sn and Ag is then only possible via solid-state grain-boundary diffusion along the Ag,Sn grain boundaries, or via (much slower) Ag,Sn volume diffusion. The observed reduction of the average IMC grain size at lower annealing temperature also corresponds to a higher number of fast diffusion paths along the IMC grain boundaries. Due to the shorter diffusion distances and the higher availability of fast diffusion paths, the TLP reaction can effectively be completed faster at lower temperature than at higher temperature, despite the lower thermal activation for diffusion and dissolution. This effect is further supported by the lateral outflow of Sn, which is more pronounced at lower temperature (also cf. discussion below). Thus, both process time and process temperature can be considerably reduced with respect to classical Ag–Sn TLP bonding[10,17,19] by the application of rapid heating. Figure 6 provides a graphical summary of the observed microstructural evolution within the bonding area.

B. Defect Generation and Joint Quality

As shown in Sections III–A and III–B, a number of defects are formed upon TLP bonding for the various process conditions (cf. Table 1 and Figure 2). The different types of defects can be classified as follows: (i) extended, mostly non-spheroidized pores just at the onset of the isothermal holding step (cf. cases 1 and 2 in Figure 2); (ii) larger spheroidized pores (case 3); (iii) small spherical pores within or in-between Ag,Sn grains (cases 1–6); and (iv) large-scale gaps (cases 4–6). The initially observed extended pores (class i) are a direct consequence of the use of interlayer foils at environmental pressure, which inevitably leads to gas entrapment upon assembly of the joining components (cf. e.g., Reference 24). In the case of slow heating to 300°C (case 3), the entrapped gas can spheroidize (class ii) once the melting Sn becomes fluid enough. Also the small spherical pores (class iii), present for all cases, can be attributed to small amounts of entrapped gas, which in this case are closed in by the growing Ag,Sn particles. By contrast, the large-scale gaps (class iv), which have a much more grave effect on the joint integrity, are formed
only upon fast heating, and are of distinctly different nature, as will be discussed in the following.

For TLP systems with IMC formation, the occurrence of large-scale defects within the bonding zone is known to be closely linked to the growth behavior of the IMC grains during the TLP process\(^6,10\): formation of Ag\(_3\)Sn from Ag and (liquid) Sn is associated with a certain volume contraction.\(^{10}\) If individual IMC particles growing from both sides into the liquid Sn impinge onto each other in an early stage of the TLP process (either upon heating,\(^6\) or during the holding step\(^{10}\)), the resulting solid bridge across the liquid-phase band will block the further compaction of the bond line by capillary action. As a consequence, the volume shrinkage upon IMC formation cannot be compensated, resulting in the presence of bonding defects in the center of the bonding zone. This problem can be mitigated by a decrease of the average IMC grain size, either by increasing the heating rate to reduce the time for grain growth upon heating,\(^6\) and/or by decreasing the annealing temperature to decrease the IMC growth rate.\(^{10}\) In the present case, faster heating indeed resulted in a smaller initial Ag\(_3\)Sn grain size, as discussed above. Annealing at a lower temperature 240 °C also lead to a smaller IMC grain size and to a more pronounced reduction of the bond thickness than upon annealing at 300 °C (cases 5 vs. 6 in Figure 2). However, the observed microstructural refinement had little effect on the amount of the critical large-scale gaps (class iv): although the vertical expanse of this class of porosity scaled with the reduced bond thickness, its lateral expanse remained similar. Accordingly, a similar average shear strength was finally obtained for both annealing temperatures.

In the present case, the emergence of the large-scale gaps is caused by another mechanism closely connected to the observed lateral outflow of liquid Sn: large-scale gaps as well as massive outflow of liquid Sn were both observed upon fast heating once the Sn was fully liquefied, but not upon slow heating. Squeeze-out of the liquefied interlayer is a typical phenomenon observed upon TLP bonding, both for foil-based approaches (e.g., Reference 19) and for coating approaches for various degrees of pressure (e.g., Reference 43). Here, the analysis of the microstructural evolution observed upon heating provides a plausible explanation: as elaborated above, the application of low heating rates stimulates the formation of large Ag\(_3\)Sn particles that are big enough to bridge the entire stretch of the interlayer and already present once the Sn interlayer is fully liquefied. The joining zone is thus stabilized against the applied bonding pressure, and virtually no lateral squeeze-out of Sn (and entrapped gas) takes place. By contrast, application of a fast heating rate suppresses the formation of larger Ag\(_3\)Sn particles upon heating, especially for the lower annealing temperature of 240 °C. Hence, vertical compression of the bonding zone and massive lateral squeeze-out of liquid Sn can occur once the interlayer is fully liquefied, despite the comparatively low bonding pressure. Simultaneous to the lateral squeeze-out of Sn, also the entrapped gas is squeezed more flatly, now forming extended gaps (cases 4 to 6) instead of spheroidized pores (case 3).

Thus, the combination of a foil-based TLP approach with fast heating eventually results in pronounced defect formation. Since the entrapment of air by the Sn interlayer foil upon assembly follows a certain arbitrariness, also the amount of gap-like defects varies from joint to joint. This results in the pronounced scatter of the shear strength values (cf. Figure 3), although the average shear strength is still comparable with values obtained by classical foil-based Ag–Sn TLP processes,\(^{10,19}\) and higher than for most modified TLP setups (e.g., References 27, 28 and 44).

Albeit generating extended bond defects, the lateral squeeze-out of Sn associated with the application of fast heating rates also brings some benefits: as discussed above, the massive lateral outflow of Sn decreases the effective amount of Sn in the bonding zone, resulting in fast completion of the TLP reaction despite the use of comparatively thick, foil-type interlayers. A secondary positive effect is the reduced amount of (comparatively brittle) Ag\(_3\)Sn phase and reduced average Ag\(_3\)Sn grain size in the final bonding zone, increasing the intrinsic strength of the bond.

C. Effect of US Application upon TLP Bonding

Application of a weak lateral US agitation of 62.5 mW/mm\(^2\) for 1 second, accurately placed after establishment of the liquid phase, lead to a pronounced enhancement of the Ag\(_3\)Sn formation (cf. case 7 in Figure 4 vs. case 2 in Figure 2), as well as to a strong reduction of bond defects immediately after US application. Hence, in comparison to the optimized TLP process at 240 °C without US support, a strongly enhanced shear strength with reduced scatter resulted (Figure 3).

For classical soldering, application of low-intensity US agitation upon solder solidification was shown to strongly improve the mechanical properties of the solder bond by modification of the solidification microstructure.\(^{45}\) In the present case, the introduction of US primarily resulted in a pronounced and much more homogeneous formation of small Ag\(_3\)Sn grains, changing from a few isolated clusters (case 2 in Figure 2) to dense Ag\(_3\)Sn bands (case 7 in Figure 4). This observation can be attributed to the mechanical agitation of the liquid Sn by cavitation and acoustic streaming.\(^{46}\) The agitation leads to an enhanced contact formation between the just molten Sn interlayer and the Ag surfaces, and promotes the initial intermixing between Ag and Sn as well as the subsequent nucleation of the Ag\(_3\)Sn particles. This effect is similar to ultrasound-assisted classical soldering used to remove oxide layers and enhance surface wetting (e.g., References 45, 47 and 48), although in the present case surface oxides play a less significant role due to prior etching.
Drastic acceleration of Ag$_3$Sn formation upon US application was also reported in Reference 31, albeit for US agitation perpendicular to the bond interface with nominally much higher power densities and longer duration.** The mechanisms leading to the acceleration of the TLP reaction as proposed in Reference 31 were (i) the removal of surface oxides by acoustic cavitation effects (see above), (ii) faster growth of the Ag$_3$Sn grains by pronounced US-induced Ag$_3$Sn grain-boundary grooving, respectively, the formation of open channels between the IMC grains, thus providing more direct contact between unreacted solid Ag and liquid Sn, and (iii) lateral extrusion of liquid Sn, thus reducing the effective bond thickness and time for Sn consumption (see above). In the present study, no clear indications for grain-boundary grooving with accelerated IMC growth were found, possibly due to the deliberately chosen low power and short duration of the US pulses; in contrast to the strongly increased density of Ag$_3$Sn grains after US application, the average grain size was not dramatically increased with respect to the case without US (cases 7 and 8 in Figure 4 vs. cases 2 and 5 in Figure 2). Increasing the length of the US pulse beyond 1 second primarily lead to a decrease of the shear strength, which can be attributed to the advanced degree of transformation after 1 second of US application (case 7 in Figure 4): since a mechanical bond between upper and lower substrate is already established, further application of the lateral, in-plane US agitation primarily acts as an unfavorable mechanical strain of the joining zone. Reduction of the bond thickness by lateral extrusion of Sn out of the bonding zone was found to be similar with or without US application, being mainly controlled by the size of the Ag$_3$Sn grains at the onset of melting (see above). Hence, similar annealing times were necessary to complete the TLP reaction for both cases.

The most beneficial effect brought forward by introduction of US in the present case is the strong reduction of bonding defects: Obviously, US agitation of the liquid Sn phase not only produces a better contact between the reaction partners, but also leads to lateral expulsion of the entrapped gas during the lateral squeeze-out of Sn. As a consequence, the formation of extended gaps within the joining zone is avoided, and the extent of well-bonded areas as well as the corresponding shear strength is significantly increased (Figures 3 and 5). This allows the foil-based TLP process to reach strength values of more than 100 MPa with strongly reduced scatter as obtained with a coating-based Ag–Sn TLP process,[17] but with a considerably shorter processing time and at lower processing temperature. Hence, the additional application of US fully eliminates the negative effects of a foil-based TLP process combined with fast heating rates. A shear strength around 100 MPa, the highest value thus far reported for Ag–Sn, may be considered as the maximum mechanical strength achievable in this TLP system. With respect to the results obtained in the present work and in Reference 17, two factors can be identified necessary to achieve this maximum value: (i) formation of a nearly defect-free bond area, and (ii) a thin bond line and correspondingly small average Ag$_3$Sn grain size (less than about 6 μm) in order to achieve a high intrinsic strength of the IMC bond line. Notably, the here employed power densities and pulse durations for US-supported TLP bonding are considerably lower compared to the previously reported approaches, and are similar to parameters as used in electronics for die attach by thermosonic flip-chip bonding.[37,49] Hence, US-supported TLP bonding with low-power US represents a promising method for manufacturing of joints with superior temperature stability and strength for electronics and other fields of application.

V. CONCLUSIONS

In the present study, the effect of fast heating and low-power US agitation on the microstructural evolution and the bond quality upon foil-based Ag–Sn TLP bonding was investigated.

- Rapid heating with 20 Ks$^{-1}$ to an isothermal annealing temperature of 240°C, i.e., slightly above the melting of pure Sn, strongly suppresses the formation of Ag$_3$Sn upon heating. Due to use of Sn foils as interlayer material with few contact points to the Ag metallization of the substrates, only localized clusters of Ag$_3$Sn are formed. Once the liquid phase is fully established upon further heating or isothermal holding, rapid nucleation and growth of Ag$_3$Sn sets in.

- The suppression of Ag$_3$Sn formation during rapid heating enables pronounced lateral squeeze-out of liquid Sn and a strong contraction of the bonding zone. Combined with gas which is entrapped between foil and substrates, this leads to the generation of extended gaps within the bonding zone, and to a large variation of the bond strength.

- Due to the refined bond microstructure upon annealing 240°C and the reduced amount of Sn in the bonding zone, the TLP reaction is already completed after 180 seconds, leading to a total process time of less than 4 minutes with an average shear strength of (58 ± 22) MPa.

- Application of a weak US pulse immediately placed after emergence of the liquid phase not only strongly enhances Ag$_3$Sn nucleation, but also eliminates the large-scale bonding defects by lateral expulsion of the entrapped air. This is reflected by a considerable increase of the shear strength to ≥ 100 MPa and a strongly reduced scatter.
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CONFLICT OF INTEREST

The authors declare no competing interest.

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