

Silver-Indium Transient Liquid Phase Sintering for High Temperature Die Attachment

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Abstract—The demand for electronics capable of operating at temperatures above the traditional 125°C limit continues to increase. Devices based on wide band gap semiconductors have been demonstrated to operate at temperatures up to 500°C, but packaging remains a major hurdle to product development. Recent regulations, such as RoHS and WEEE, increase the complexity of the packaging task as they prohibit the use of certain materials in electronic products such as lead (Pb), which has traditionally been used in high temperature solder die attach. In this investigation, an Ag-In solder paste is presented as a die attach alternative for high temperature applications. The proposed material has been processed by a transient liquid phase sintering method resulting in an in situ alloying of its main constituents. A shift of the melting point of the system, confirmed by differential scanning calorimetry, provided the basis for a breakthrough in the typical processing temperature rule. The mechanical integrity and reliability of this novel attachment material is discussed.

Keywords—Die attach, DSC, high temperature, reliability, solder joint, transient liquid phase sintering

INTRODUCTION

The development of electronics and microsystems that can operate at temperatures in excess of the traditional maximum of 125°C [1] is a critical enabling technology for the creation of next generation electronic systems for a wide range of military and commercial applications including avionics, hybrid-electric automotive electronics, deep well drilling, chemical processing systems, and space/earth exploration. Critical elements of these systems are the subassemblies for power control, distribution, and management. The last several years have seen the advent of silicon carbide (SiC) devices operating at temperatures well above 125°C [2]. These devices provide higher switching speed and lower on-state losses with higher thermal conductivity when compared with silicon. Developing reliable technologies for packaging is now the main hurdle to successful operation of SiC based electronics at high temperature.

This work focuses on the first-level interconnection process known as die attach, the primary function of which is to

mechanically secure the semiconductor chip to a lead frame or substrate, and to ensure it does not detach or fracture over an operational lifetime that may include power and temperature excursions. One of the most common approaches for packaging power semiconductor devices is to mount the back of the chip on a ceramic substrate with a suitable die attach. However, unique materials and manufacturing processes are required for die attachment at high temperatures.

When metallic solders are used for die attachment, the application temperature (T_a) (i.e. the temperature at which the system will operate) dictates material selection and its accompanying assembly process. This restriction arises due to the nature of the reflow bonding mechanism, where the attach material has to be melted before resolidifying to form a reliable metallurgical interconnection. This implies a design rule where the melting temperature (T_m) of the attach material needs to be higher than T_a , and the processing temperature (T_p) above T_m ; [$T_p > T_m > T_a$]. The processing temperature refers to the peak temperature required during the reflow process. Residual stresses are built into the system when cooling from extreme processing temperatures, often leading to reliability problems associated with thermally activated failure mechanisms [3]. An alternative for the above temperature hierarchy rule is needed for the advancement of the high temperature die attach process, where increasing application temperature is dictating the development of new methodologies. Sintering of silver flakes has been suggested as an alternative [4]. This low temperature joining technique is based on the concept of diffusion welding, in which the material is subjected to a temperature below its melting point, and bonding is assisted by application of external pressure during processing. However, pressures of up to 40 MPa are required to form a reliable joint, frequently causing catastrophic damage to the devices. A pressureless alternative had been investigated [5, 6] using a silver nanoparticle solder paste. This technique utilizes a reduction of total free energy as the driving force for the sintering reaction. However, this approach has been shown to have reliability limitations [7].

A transient liquid phase sintering process (TLPS) is presented in this paper as a viable alternative for high temperature die attach. Powder mixtures consisting primarily of a high melting point base metal (constituent B) can be more easily sintered when combined with a low melting point additive (constituent A). The TLPS of powder mixtures depends on the formation of a liquid phase of constituent A at a processing temperature (T_p) that is below the melting point of the base metal powder (T_{mB}),

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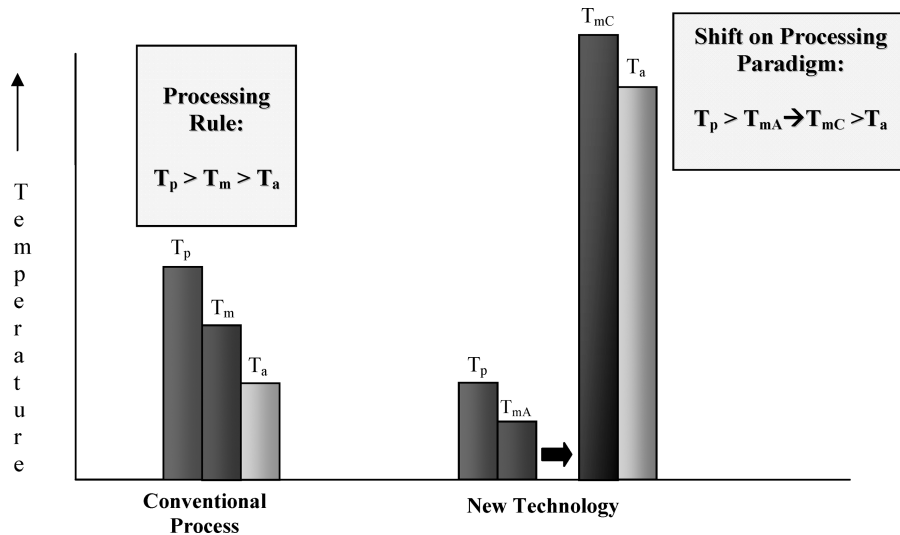


Fig. 1. Graphical representation of the conventional process vs. the TLPS technique.

yet above that of the additive phase (T_{mA}). The presence of the liquid enhances mass transport rates and densification (compared to solid-solid sintering) by exerting a capillary pull to rearrange powder particles [8, 9]. This liquid isothermally solidifies over time (transient process) by solute diffusion into the base metal particles as the T_p is held above T_{mA} . The advantages offered by TLPS over conventional solid state sintering include: rapid densification during sintering because of capillary action, enhanced mass transport created by the liquid phase, and lower sintering temperatures. The biggest advantage of TLPS is the fact that the liquid solidifies isothermally, resulting in an in situ alloying process that yields an alloy having an equilibrium bulk composition that corresponds to the initial mixing ratio of its elemental constituents. This new alloy exhibits a higher melting point (T_{mC}) that permits application temperatures well above the processing temperature.

In this investigation, an Ag-In solder paste is presented as a high temperature die attach alternative. This material overcomes the traditional temperature hierarchy paradigm by permitting solder attachment at temperatures below the final melting point of the newly formed alloy. This enhanced temperature rule is given by: $T_p > T_{mA}$ which transforms into $T_{mC} > T_a$, where T_a is, in the end, constrained by T_{mC} , which can be much higher than T_{mA} . Fig. 1 shows a graphical representation of the concept. The processing temperature (T_p) is defined by the lower T_{mA} , resulting in $T_m/T_p > 1$, therefore, providing a method to have a relatively low processing temperature while resulting in a high temperature resistant material. In this case, indium constitutes the A material and silver constitutes the B material. The mechanical integrity of this material will be investigated by die shear testing, whereas its long term reliability will be assessed via passive thermal cycling. The thermal stability of this novel system will be studied by differential scanning calorimetry (DSC).

EXPERIMENTAL PROCEDURE

A. Solder Paste

Solder paste is a viscous mixture of solder powder and flux. The viscous nature of the paste allows it to be stencil printed or

dispensed, thus enabling the implementation of a high throughput production practice. When compared with thin film planar approaches, the paste advantage is twofold; it provides a less expensive alternative readily available for the practitioner, and a faster time to solidification. The particle size of the solder powder is dictated by the end application, with smaller particle sizes used for smaller depositions. The flux serves two functions. First, during soldering it removes metal oxides from the surfaces to be joined; and second, during printing it is a suspending vehicle for the powder. The rheology of the flux vehicle must provide a stable suspension for the particles not only during storage and handling, but also during the assembly process. With properly formulated solder paste, the material can be fairly homogeneous, thus allowing the composition of the mixture to be consistent during its deposition.

For this study a solder paste consisting of elemental silver and indium particles was formulated. The metallic composition of this paste was fixed at 70 wt.% silver, 30 wt.% indium in a no-clean rosin mildly activated (RMA) flux. Elemental composition of the paste was based on the phase diagram where the solid solubility of indium into silver will permit complete diffusion without the risk of a persisting low temperature phase. The particle shape and size was optimized for the die attach process under investigation. Details of the effects of paste composition and particles size are given elsewhere [10].

B. Fabrication

Specimens for mechanical and reliability testing were fabricated by stencil printing the solder paste followed by die placement; the transient liquid phase sintering process completed the assembly sequence. Silicon chips, of various sizes, were attached to ceramic direct bond copper (DBC) substrates using the above assembly sequence; details regarding the test specimens are given in Table I.

The solder paste was transferred to the ceramic substrate using a stencil and a metal squeegee. Stencil apertures were laser cut in a 200 μm thick foil, to match the three die sizes. Fig. 2 shows an image of the stencil.

Table I
Specifications for Test Specimens

Die		
Material	Size	Thickness
Si	3.3 mm × 3.3 mm	500 μm
	5 mm × 5 mm	
	7.5 mm × 7.5 mm	
Substrate		
Material	Metallization	
AlN or Al ₂ O ₃	DBC (Direct Bond Copper)	
	Ni—Barrier Layer	
	Ag—Bonding layer	

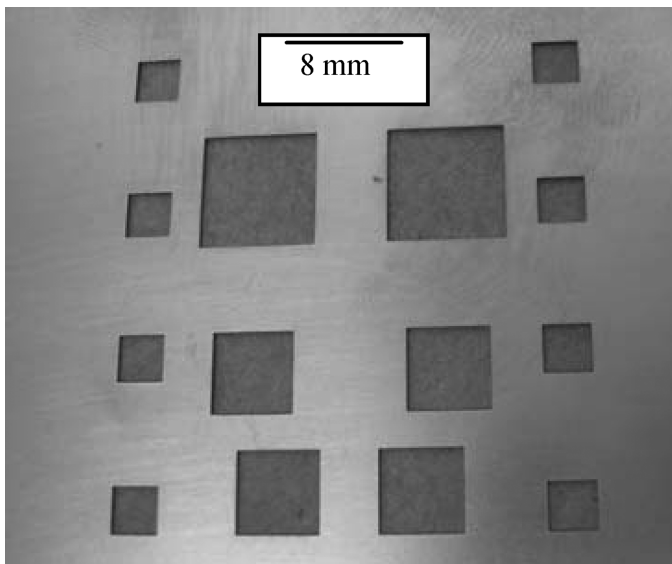


Fig. 2. Laser cut stencil used for paste deposition.

Fully populated substrates were processed in a furnace following an optimized thermal profile, in which the heating rate (60°C/min.), processing temperature (300°C), and isothermal holding time (40 min.) were defined based on the kinetics of this particular binary system [10]. It has been demonstrated [11] that the achievement of a shift in the melting point of the system is a complex problem governed by a multistage diffusion controlled mechanism. Depending on the solder paste composition, particle size, and maximum allowable processing temperature, a thermal profile can be tailored to obtain the desired shift in melting point. In principle, higher processing temperatures result in shorter isothermal holds. In this case, samples were processed at 300°C and held for 40 min. Fig. 3 depicts an as-built, fully populated substrate with the three die sizes. The as-built bond line thickness was measured to be 100 μ m, that is, a 50% reduction from the initial deposition.

C. Shear Testing

The mechanical strength of this attach system was assessed by means of a standardized die shear test. This destructive test

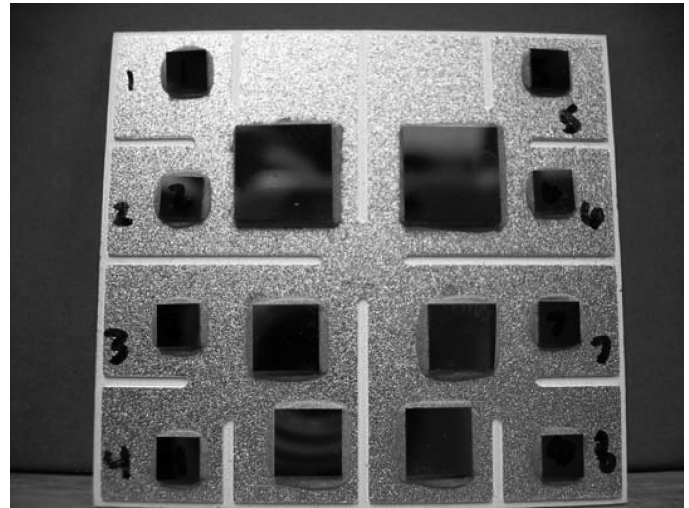


Fig. 3. Fully populated DBC substrate fabricated with the TLPS process. The three die sizes are illustrated.

measures the maximum shear load to failure. The fractographic analysis of the surfaces provided insights into the fracture mode and possible failure mechanism. The die shear test was performed according to the MIL-STD883G Method 2019.7 [12]. For this investigation the equipment and test parameters were as follows: a Dage 2400 shear tester was used with a DS 20 Kg-f load cell, and the test was set as destructive at a test velocity of 200 μ m/s and a test height of 200 μ m.

D. Reliability Assessment

The ability of this material to render its intended function throughout its expected life cycle was studied by means of passive thermal cycling. When test specimens are subjected to thermal cycling, the mismatch in coefficient of thermal expansion between the substrate and die will cause stresses to be generated in the attach material. By subjecting a statistically significant number of samples to different temperature cycles (given by ΔT), distinct values of strain range are developed. Two different accelerated thermal profiles were generated for this investigation. The high temperature profile (HT) was designed to subject the samples to temperature cycling from -55°C to 185°C with a 5 min dwell at -55°C and a 10 min dwell at 185°C for a ΔT of 240°C at a T_{mean} of 65°C . The low temperature profile (LT) was designed to subject the samples to temperature cycling from -55°C to 150°C with a 5 min dwell at -55°C and a 10 min dwell at 150°C for a ΔT of 205°C at a T_{mean} of 47.5°C . The combination of the three die sizes with the two thermal profiles (HT and LT) provided a total of six strain range levels. Samples were taken out of the temperature cycling chamber after every 50 cycles for inspection using x-ray imaging. Failure of the die attach was defined based on crack propagation through the bond area, as observed by x-ray. Joints were deemed failures when the crack/delamination exceeded 20% of the bonding area. Initial characterization of the as-built samples was used as the baseline for failure definition.

E. Differential Scanning Calorimetry (DSC)

The amount of solute mass (indium) that has diffused over time into the base metal (silver) defines the progression of the

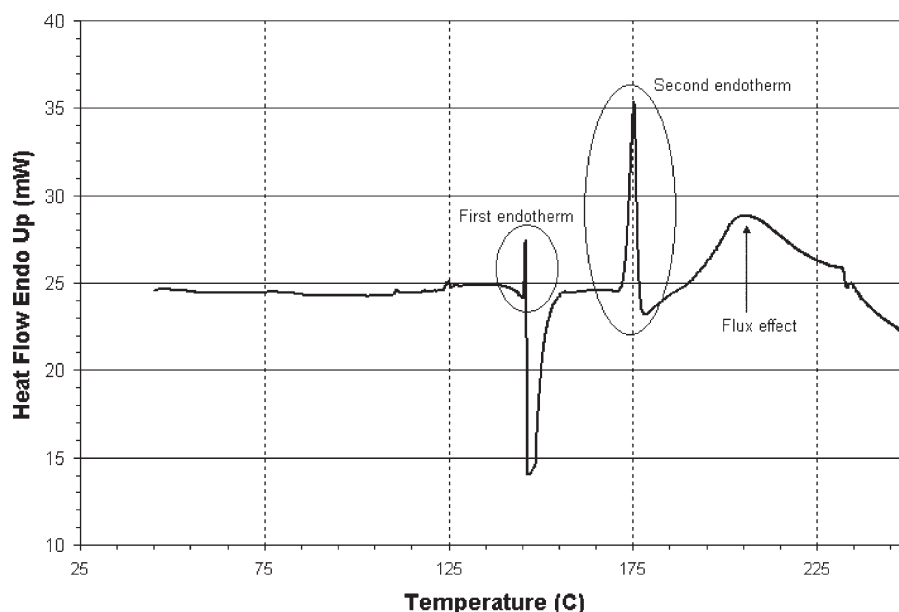


Fig. 4. Typical DSC trace for Ag-In paste sample heated to 250°C. Endotherms are shown as upwards peaks.

process. The problem of inaccurate measurement techniques for quantifying the kinetics of this isothermal solidification reaction has been addressed by the development of a new technique using differential scanning calorimetry (DSC) [13]. This method allows the determination of the amount of initial liquid formation as well as its duration, that is, the kinetics of the isothermal solidification process. The DSC measures heat flow (mW) of a sample relative to a reference cell throughout a specified profile in which the temperature, heating/cooling rate, and isothermal holds can be controlled. The progression of the TLPS process along its stages can be accurately calculated using this technique [14]. A Perkin Elmer DSC Pyris 1 was used in conjunction with the aluminum sample holder pan kit (PE Kit: 219-1073). Samples consisted of 30–80 mg of the solder paste placed in the aluminum pan according to the standard operating procedure of the equipment; the exact weight of each sample was measured and recorded. A baseline test using empty reference samples was conducted prior to the testing and subtracted from the final data. This accounted for any artifacts introduced by the equipment itself. The equipment was calibrated using a standard indium sample as specified by the supplier. A nitrogen purge was used to avoid oxidation during the test.

RESULTS

The advantage of the Ag-In TLPS joint relies on the fact that the processing temperature (T_p) is dictated by the melting point of indium (156°C) [15]. Isothermal holding at the selected T_p permits solute diffusion into the silver base metal. This mass transport mechanism is the basis for the solidification of the In-rich liquid that culminates in the in situ alloying of its constituents. This dynamic process is driven by the indium concentration gradient across the solid-liquid interface. The kinetics of formation are discussed in detail elsewhere [16]. The successful implementation of this material for high tem-

perature applications depends on the ability to transform the In-rich liquid into a higher melting point solid. The presence of a fraction of persistent low melting In-rich liquid may jeopardize the ability of this material to survive high temperature applications.

An experimental method to study the kinetics of the TLPS process has been presented by Corbin [17], in which the thermal data from a differential scanning calorimeter (DSC) trace is used to calculate the extent of the reaction. Melting a crystalline material requires an energy input that is proportional to the mass of the phase being transformed. This energy is recorded in the DSC as an endothermic (upward) peak at the transformation temperature. The reverse of the above reaction is solidification; upon cooling below the transformation temperature, energy is released by the system. The amount of released energy can be obtained from the area of the exothermic (downward) peak. This energy is proportional to the amount (mass) of the solidifying material.

Heating a sample of the solder paste to the processing temperature resulted in the DSC trace shown in Fig. 4.

During this heating cycle, two endothermic peaks that corresponded to phase changes were observed (these reactions were confirmed with the phase diagram) [15, 18]. The first endothermic peak corresponded to the eutectic reaction at 147°C [18], ϕ (66 at.% In) + In (100 at.% In) \rightarrow L (97 at.% In); whereas the second endotherm occurred at 170°C where a peritectic reaction [18] [ϕ (66 at.% In) \rightarrow γ (33.5 at.% In) + L (96.2 at.% In)] takes place. The formation of the In-rich liquid via the eutectic reaction depends on the initial extent of material with the eutectic composition. It has been reported [19] that this eutectic could be formed at room temperature as silver and indium are mixed together; its formation is accelerated even further with temperature. The initial mass fraction of this phase is minimal and it is strongly dependent on the heating rate used to reach the processing temperature [20]. Immediately after formation, the In-rich liquid infiltrates the

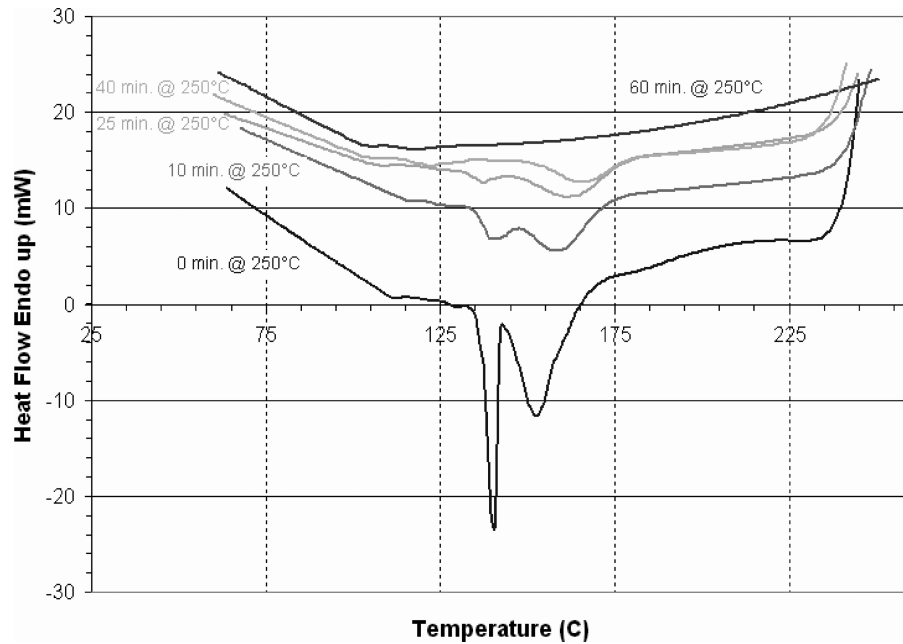


Fig. 5. DSC traces for the cooling stage of samples treated at 250°C for various holding times. Exotherms are given by the downward peaks. It was observed that at 40 min, the eutectic reaction at 147°C was no longer detected; whereas at 60 min no exotherms were recorded. (These traces do not correspond to the heating curve given in Figure 4).

silver powder compact by capillary action inducing an erosion effect as it comes into contact with the silver solid. At a temperature between 147°C and 170°C the newly formed liquid coexists with ϕ , this ϕ phase is an In-rich material that is stable from room temperature up to 170°C [18]. This L + ϕ is a stable system in the temperature range between 147°C and 170°C, and evidence of this stability can be seen in Fig. 4 where energy was released (exotherm) following the eutectic reaction. Continuous heating toward the processing temperature results in the peritectic reaction at 170°C, where ϕ melts, resulting in more In-rich liquid. As can be observed, both of these reactions contributed to the formation of In-rich liquid during the heating stage. Upon cooling, after insufficient isothermal holding time at T_p , the fraction of material with the In-rich composition(s) underwent solidification reactions that are shown in Fig. 5.

For short holding times, an exothermic reaction at 170°C (Note: undercooling was observed on all samples) resulted in the precipitation of primary ϕ while the remaining fraction of eutectic liquid transformed into eutectic solid (ϕ + In). As the holding time increased, more diffusion of indium into silver occurred, resulting in the isothermal solidification of the In-rich liquid. From the DSC traces in Fig. 5 it was observed that the magnitude of the eutectic exotherms decreased with holding time up to 40 min. At holding times longer than 40 min, the In-rich liquid solidified as ϕ with no eutectic transformation. When the holding time was even longer (≥ 60 min permitting complete isothermal solidification), all of the In-rich liquid transformed into the Ag-rich solid solution. No exotherms were observed during the cooldown. At this point, a newly formed in situ alloying process had been accomplished with a resulting high melting temperature Ag-rich material, which based on the phase diagram, should exhibit a

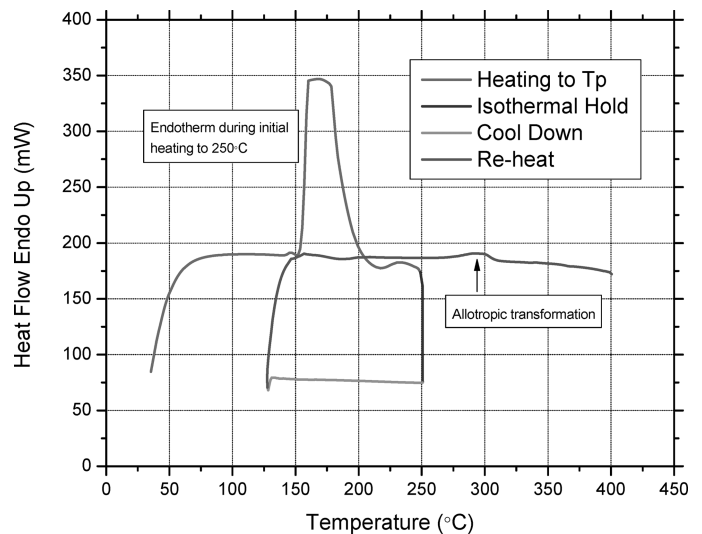


Fig. 6. DSC trace for the heating-cooling-reheat cycle used to demonstrate the thermal stability of the newly formed material.

melting point above 650°C. To validate the thermal stability of this newly formed alloy, a sample was subjected to a second heating excursion that simulated an application condition of 400°C. In Fig. 6, a DSC curve of a sample initially heated to a T_p of 250°C and then held for 60 min is given.

During this first heating stage, the endotherm associated with the melting of the In-rich phases was observed. Upon cooling to 125°C, after a 60 min hold, no exotherms were observed. This suggested that the diffusion controlled transformation had been successfully achieved. Heating the sample to 400°C resulted in no new endotherm, confirming that the material was

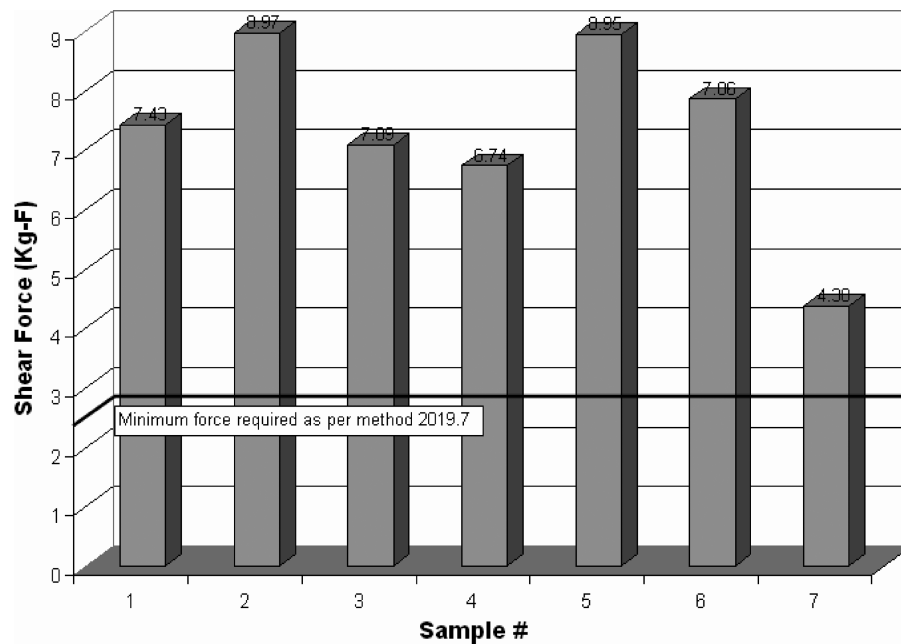


Fig. 7. Shear strength data for 3 mm square dice attached with the Ag-In TLPS process.

stable up to such temperature. From the DSC trace, a small endotherm was observed at 300°C corresponding to an allotropic transformation of the Ag-rich phase γ to the HCP ζ phase (this is a solid to solid transformation).

The purpose of the die shear strength test is to determine the integrity of the material and the procedure used to attach the die to the substrate. This determination is based on measuring the force required to cause failure in conjunction with a qualitative description of the fracture surface. The shear strength of an attachment material is reported as the force required to shear the die from the substrate; values below the specified (Fig. 2019-4 in Ref [12].) minimum constitute a failure. Results from this test must include force data together with the separation category, that is, a description of the physical appearance of the specimen. For this investigation, a total of seven 3 mm \times 3 mm dice were tested using the equipment and settings described previously in Section C. Quantitative results are given in Fig. 7, where the minimum force required for a 3 mm square die is given by the horizontal line at 2.5 Kg-f.

Shear strength data for the proposed attach material/method fulfills the minimum requirements from the standard, with all samples above 4 Kg-f. The experimental data can be summarized as having mean shear strength of 7.3 Kg-f. All samples, except sample 5, failed cohesively through the attach material, suggesting acceptable adhesion to both the die and substrate. Sample 5 failed through the attach as well, although some residual silicon was left on the surface. Evidence of this can be observed in Fig. 8. As stated in the standard [12], residual silicon material attached in discrete areas of the die attach medium shall be considered as evidence of adhesion.

A total of 2,200 LT cycles were completed for all three die sizes without observing discernible degradation of the attach

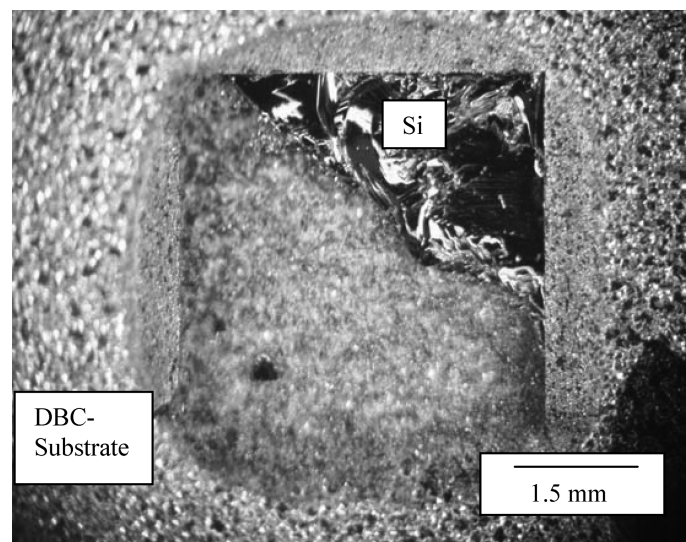


Fig. 8. Sample 5 fracture surface after the die shearing test. The attach material on the DBC substrate together with residual Si on the surface served as evidence of good adhesion.

material as revealed by x-ray and physical inspection. For the HT profile, no failures were observed after 1,000 cycles for all sizes, though extensive damage to the DBC substrates was observed. Separation of the copper from the ceramic material started at the corners of the substrate, and fracture progressed as a crack through the ceramic. From these observations it was revealed that the proposed Ag-In TLPS attach material outperformed the DBC substrates during the thermal cycling test. Results obtained from life cycle data (cycles), were comparable to the observed performance of the Pb-rich solder, the most widely used material for high temperature electronics

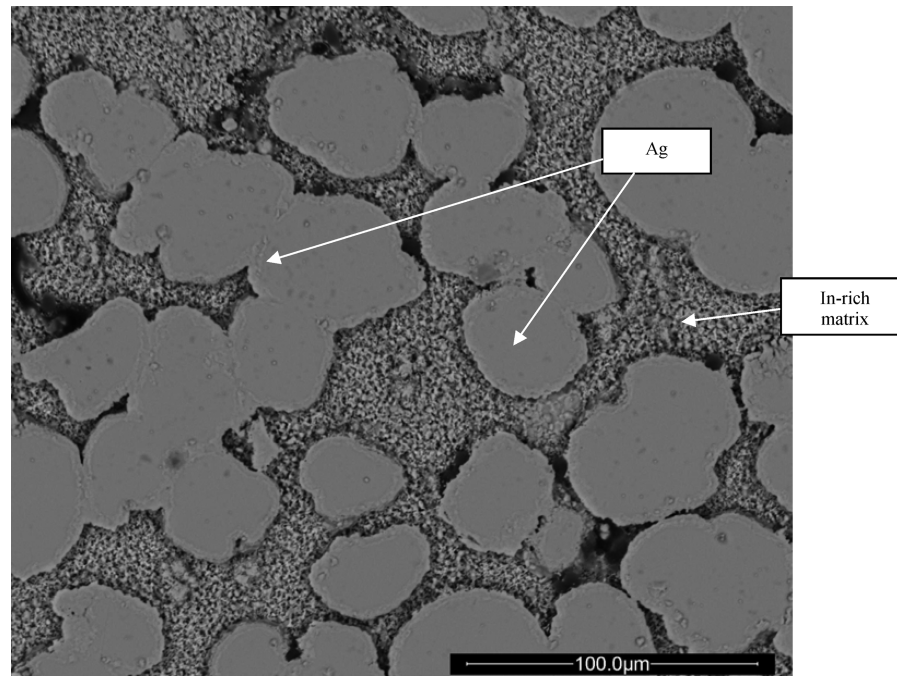


Fig. 9. BSE image of a 70 wt.% Ag, 30 wt.% In specimen heated to 250°C and cooled to room temperature with no isothermal holding.

applications [7, 21]. When compared with lead-free alternatives, it outperformed technologies such as sintered Ag-nanocolloids as well as sintered Ag nanoparticle pastes [7, 21]. In the formulations studied, both of these technologies resulted in complete separation of the die from the substrate. For the case of the LT test, all samples failed at or before 700 cycles, whereas for the HT condition all samples failed at or before 400 cycles. From this reliability assessment it was concluded that the structure of this TLPS material provided a compliant attach capable of absorbing the strain energies associated with the thermomechanical deformations. The holding time at a processing temperature provided enough energy to reach a full phase transformation so that equilibrium or near-equilibrium conditions resulted. The stability of this system makes it less sensitive to future thermal excursions or high temperature storage conditions.

DISCUSSION

Transient liquid phase sintering is a powder metallurgy method in which the presence of a melting point depressant, indium in this case, allows for a relatively low temperature assembly process. In situ alloying during the isothermal solidification stage results in the formation of a material with physical and mechanical properties close to that of the parent material, silver in this instance. Because TLPS is a powder metallurgy technique, it is expected that the final structure will be porous or cellular in nature. External pressure has typically been used to enhance the densification of these materials. In the current investigation, a pressureless system was developed in which the wetting liquid served as the driver for densification. Fractional densities ranging from 75–95% have been reported in the literature [8] when this technique is used. The formation of this cellular structure

dominates the mechanical, electrical, and thermal response of such materials [22, 23] where the concept of microstructure no longer refers only to the grain scale but also to size, volume fraction, and shape of the pores. It has been shown that the fractional density of the proposed material is strongly correlated to the volume fraction of the melting point depressant constituent [10]. In this study, a material with a 30 wt.% In (38 vol.% In) was used. A resulting fractional density of $88\% \pm 1.43$ (vol.%) was measured using a point count method technique [16].

Samples of the 70 wt.% Ag, 30 wt.% In paste treated for varying holding times were epoxy mounted, polished, and etched in order to elucidate the metallurgical reactions taking place during TLPS as well as to confirm DSC results. The microstructure developed in these samples was observed using backscattered electron (BSE) imaging in the ESEM. An EDS analysis was also performed to identify the phases present by their elemental composition. Fig. 9 shows a BSE of the microstructure of a sample heated to 250°C at 60°C/min with no isothermal holding time.

The presence of the low melting point, In-rich lamellar structure of the eutectic solid is evident. It is observed that this phase constitutes the continuous matrix where the silver particles are embedded. EDS analysis revealed that the matrix is composed of eutectic material and ϕ (67 at.% In). Fig. 10 depicts the microstructure of a sample treated at 250°C for 60 min. In this case, the γ phase is evenly dispersed through the matrix. This represents the homogenization stage that tends to the composition corresponding to final equilibrium. At this stage of the process, all the indium is present in the form of a high melting point $\alpha + \gamma$ microstructure. This was confirmed (Fig. 5) by the absence of exothermic peaks during cooldown. The formation of this alloy provides the fundamental explanation for our proposed material/process.

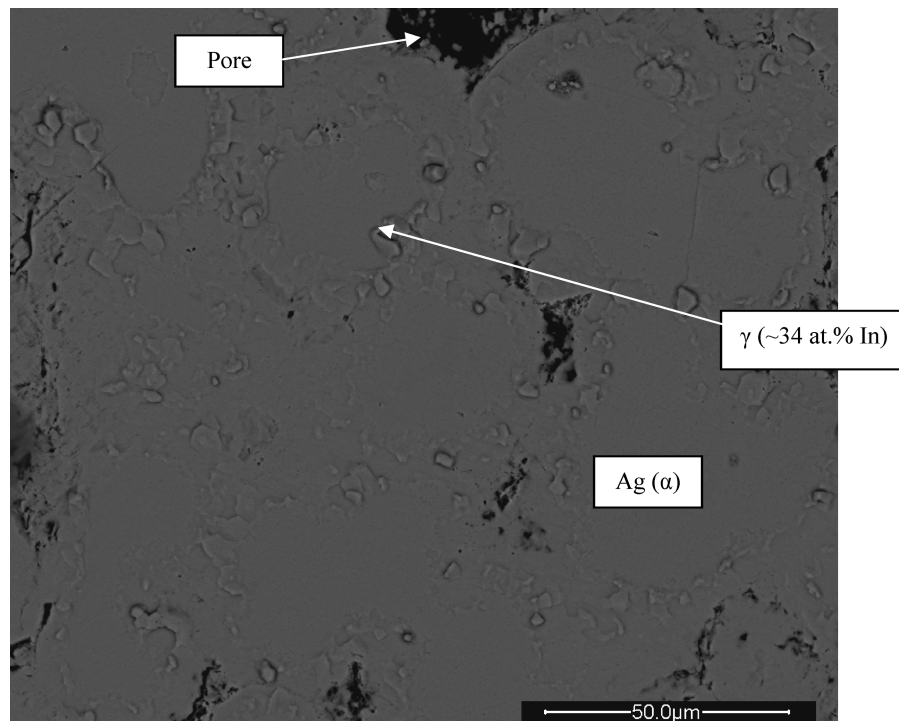


Fig. 10. BSE image of a 70 wt.% Ag, 30 wt.% In specimen heated to 250°C, held isothermally for 60 min, and cooled to room temperature.

CONCLUSION

During this investigation a transient liquid phase sintered Ag-In solder paste was presented as an alternative die attach material for high temperature applications. A fabrication process using standard stencil printing and a TLPS technique was introduced. Processing at a temperature of 250°C yielded a system with a thermal stability up to 400°C or above. A breakthrough in the typical process was established, where the reflow rule of $T_m/T_p < 1$ has now been substituted by $T_m/T_p > 1$. Both the mechanical integrity and the reliability of this system were demonstrated to be above the acceptable limits.

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