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Customized Glass Sealant for Ceramic Substrates for High Temperature Electronic Application

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Abstract

This study investigates the ceramic to ceramic bonding, using composite glass frit as the binding layer that is able to tolerate a high temperature environment for ruggedized microelectronic applications. Shear strength measurements were carried out at both ~~ambient-room~~ and high temperature (i.e. 250°C) to evaluate room and high temperature performance of the joints. The glass joints in both the Al₂O₃/glass/Al₂O₃ and AlN/glass/AlN systems maintained their integrity even when shear-tested at 250°C. The results of the mechanical and microstructural characterizations demonstrate that Bi-based two phase glass frit bonding is an effective ceramic bonding method for harsh-environment electronic packaging.

Keywords: Harsh environment; electronics packaging; ceramic substrates; Pb-free glass frit; high-temperature reliability.

Introduction

Currently industries are facing reliability issues in terms of the survivability of their Pb-free electronic systems operating in high temperature, pressure, radiation and corrosive environments [1]. In order to implement practical high-temperature electronics packaging, innovative

packaging materials and package designs concepts are required [2]. For example, it has been reported recently that the harsh environment electronics should have low power devices that should not generate much heat and it should be necessary to prevent heat transfer from the environment to the device chip as well [3]. The unavailability of a mature packaging technology for 300°C–600°C operation partially hinders development and demonstration of electronic devices for this temperature range [4]. The major challenges to realizing such a packaging technology are chemical, physical, and electrical stability of the packaging materials themselves as well as the interfaces between these materials [5].

For highly reliable packages in which excellent thermal stability and impermeability are essential, ceramic-based packages are preferred over plastics [6-8]. The demand for ceramic packages has increased a lot recently for the high performance hermetically sealed devices such as MEMS, sensors, microwave devices, and imaging devices [9, 10]. Alumina (Al_2O_3) and aluminum nitride (AlN)-based ceramics are used in many special applications where density, high hardness, chemical inertness, low thermal conductivity and good high temperature properties are required [11-13]. These excellent thermo-mechanical properties of ceramic materials, as compared to those of conventional metals, suggested the employment of ceramics in high-temperature environments.

Joining is a critical enabling technology, essential to widespread use of ceramics in many applications. Among the joining techniques, adhesive bonding is unadvisable due to the high service temperature. The high preparation cost and low efficiency limit the application of diffusion bonding [14]. Due to its simplicity and low preparation cost, brazing is recognized as the main method for joining ceramic materials [15]. However, the brazing requires high temperature (i.e. around 900°C) that will definitely destroy the electronic device functionality.

Concurrently, the large differences between ceramic materials and brazing alloy, such as coefficients of thermal expansion (CTE) and Young's modulus (E), led to high residual stresses in the joint during the cooling from the brazing temperature [16]. Besides the CTE match requirement for obtaining a hermetic seal that would withstand thermal cycling, high temperatures significantly promote thermal processes such as diffusion and degassing at material surfaces that would likely lead to contamination of the hermetic cavity atmosphere over time. Achieving and verifying hermetic seal for long-term 300°C–600°C operation will be challenging.

Package encapsulation is a critical first layer protection against external environment. In deep-hole drilling applications, it is necessary to shield circuitries from high surrounding temperature and pressure. Conventional epoxy encapsulation would not suffice in high temperature and high pressure environments [17]. Thus, an alternative ceramic encapsulation method has been designed for this study. The primary objective of this work is to develop a joining material that can be able to sustain a certain amount of pressure applied at the high service temperature. The functions of joining material are depicted in Fig. 1. A typical ruggedized electronic packaging design considers circuit components mounted on the ceramic base and has a ceramic lid which covers the components (Fig. 1). Regarding chemical compatibility between ceramic and glass materials, the use of glass frit for ceramic to ceramic bonding can be considered as a simple and yet robust method for hermetic sealing of microelectronic packages. From the previous study, it was demonstrated that the commercial available glass paste bonding was an effective die-attach method [18, 19]. The current work investigates the feasibility of using glass frits as a bonding material on bare ceramic substrates for high-temperature application, without any metallization preparation for bonding surfaces. The study focuses on mechanical and microstructural

characterizations of the glass frit joints with two different ceramic substrates. In addition, the influence of aging at 300°C on the mechanical properties of the bond was also investigated. Effects of joining temperatures on joint properties and interfacial microstructure evolution are presented.

Experiment

A mixture of commercially available two types of Bi-based glass frits and no clean flux was used as the joining material for the ceramic substrates. The glass frits were collected from a local agent of International Laboratory USA (IL), China. As per the supplier, the D_{50} of both the glass powders were around 0.9 μm (i.e. 50% of the particles in the sample are larger than 0.9 μm , and 50% smaller than 0.9 μm). The chemical composition of the glass powders are presented in Table I. The softening temperatures (T_s) of the two glass systems were around 350°C and 650°C, respectively. The recommended temperature for bonding the ceramic substrates with the low T_s (i.e. 350°C) glass frit is 430°C as per the manufacturer. The coefficient of thermal expansion (CTE) of low T_s glass frit and high T_s glass frit were $9.0 \times 10^{-6}/^\circ\text{C}$ and $7.8 \times 10^{-6}/^\circ\text{C}$, respectively. In this study, two ceramic substrates, Al_2O_3 and AlN , were purchased from Semiconductor Wafer Inc., Asia. The surface roughness of both the ceramic substrates was quite similar (i.e. $r_a=0.2$ to $0.35 \mu\text{m}$). The coefficient of thermal expansion (CTE) of Al_2O_3 and AlN were $8.0 \times 10^{-6}/^\circ\text{C}$ and $4.6 \times 10^{-6}/^\circ\text{C}$, respectively.

The mixed glass frit paste was applied in between two 0.76mm thick ceramic substrates. The paste consisted of 88 wt.% solids and 12 wt.% no-clean flux. The solid portion contained 85 wt.% of low T_s glass frit (i.e. high Bi containing frit) and 15 wt.% of high T_s glass frit (i.e. low Bi containing frit). Glass paste bonding consists of two main steps: screen printing and sintering.

Using a stainless steel stencil, the paste was dispensed on the 5 mm X 5 mm substrates and then 2.5 mm X 2.5 mm substrates were placed on them. A Nabertherm (model: N 7/H, Germany) resistance heating box furnace was used to perform the bonding process. The furnace was calibrated with a tolerance of less than $\pm 3^{\circ}\text{C}$. Fig. 2 shows the temperature profile for the bonding. During the heating cycle, the samples were first heated up to 125°C and held for 10 minutes. After heating to 250°C with a dwell time of 30 minutes in air to burn out the flux components, the paste was further heated to a peak temperature (i.e. 420°C - 440°C) for 10 minutes without external pressure to sinter the glass frit, after which the paste was consolidated into a strong and uniform bonding layer. The bonding arrangements for the two types of ceramic plates are shown in Fig.3a. The schematic of the bonding mechanism is shown in Fig.3b. After the bonding process, the samples were inspected with a microfocus X-ray inspection system (Shimadzu SMX-2000EX) to examine the voids present within the joint. The ceramic/ceramic joints were also aged at 300°C in air for up to 500 hours for reliability assessment.

To investigate the microstructure, the samples were mounted in epoxy after bonding. The samples were ground, polished and coated with a thin layer of Ta for SEM examination. The microstructural analyses of the Ta-coated cross-sectioned samples were obtained using a Philips XL 40 FEG (Philips Electron Optics, The Netherlands) scanning electron microscope (SEM). Fig. 3c shows a typical glass frit joint structures with the AlN substrates. It was found that the thickness of the joint varied between 25-40 μm for all the cases. The variation in thickness of the dispensed double phase glass frit joint was due to manual screen printing method.

Shear test was performed on bonded samples using a Dage Series 4000 Bond Tester. A shear tool height of 100 μm and shear speed of 100 $\mu\text{m/s}$ was used. The shear test was carried out at

both room temperature and at high temperature (250°C). For the high temperature shear measurement, the sample stage was heated to 250°C and each sample left on the sample stage for 15 minutes to reach the equilibrium temperature prior to the test. A total of 20 ceramic/ceramics joints were sheared for each condition.

3. Results and Discussion

X-ray inspection (μ CT) was performed on all the bonded samples to visualize the quality of the joints after bonding. This μ CT examination allows visualization of the largest pores in three dimensions and provides information about their interconnectivity in the structure. It is important to note that the X-ray images revealed a large number of voids in the glass bond for both the $\text{Al}_2\text{O}_3/\text{glass}/\text{Al}_2\text{O}_3$ and $\text{AlN}/\text{glass}/\text{AlN}$ systems as shown in Fig. 4. These large voids were generated due to coalescence of small pores during liquid phase sintering of the glass matrix. In addition the expansion of the trapped gas (i.e. the gas evolved from the decomposition of the flux) at the peak temperature also played a major role.

Fig. 5 shows the average bonding strength of $\text{Al}_2\text{O}_3/\text{glass}/\text{Al}_2\text{O}_3$ and $\text{AlN}/\text{glass}/\text{AlN}$ joints at different bonding temperatures. It is found that $\text{Al}_2\text{O}_3/\text{glass}/\text{Al}_2\text{O}_3$ showed better shear strength than that of the $\text{AlN}/\text{glass}/\text{AlN}$ joints. At 420°C, the shear strength was the lowest for both the systems. Considering the measurements deviation, the joint strengths at 430°C and 440°C are comparable for the individual systems. That means, beyond 430°C there was no significant impact of bonding temperature on the joint strength for both Al_2O_3 and AlN systems. Surprisingly, the shear strength measured at high temperature (at 250°C) showed an increase for both Al_2O_3 and AlN systems.

It is believed that some sort internal stress of the glass might arise within the glass volume due to the difference in cooling rate of the glass compositions. However, it is established that at the annealing point of the glass, stresses within the glass can relax rapidly [20]. In this case, 250°C might be very close to the glass annealing point of the particular glass matrix. Thus, even though 250°C is not that close to the T_s (~350°C) of one of the composition of the glass frit, it might have induced low temperature annealing effect to the joint, thus improving the bond strength at 250°C.

Another thing was that the thickness of the joint varied between 25-40 μm for all the cases. Definitely there would be some effect of bond thickness on the joint strength. It is believed that the more the joint thickness the more will be the amount of defects in the glass joint. Also, a thin joint is preferred over a thick one, since the stress concentration at the corners of the joint is lesser in a thinner joint. It is expected that the joint strength may be decreased with higher bond thickness. The variation in thickness of the dispensed glass frit joint was due to manual screen printing method. The comparison made within the results of the shear strength for different joint systems would be more certain if the glass bond thickness within the joint were made evenly.

The successfully joined samples were further heat-treated and characterized. Both $\text{Al}_2\text{O}_3/\text{glass}/\text{Al}_2\text{O}_3$ and $\text{AlN}/\text{glass}/\text{AlN}$ joints sintered at 430°C were aged at 300°C for up to 500 hours and their shear strengths are shown in Fig. 6(a) and (b), respectively. It was found that the shear strength did not decrease with aging. During aging any sort of deterioration is caused by the change in the interface (e.g. reaction in metal/metal joint in solder joint upon aging at the interface). The current glass/ceramic interface is more or less inert at 250°C. That's why there was no deterioration observed during aging for 500 hours. In fact, a slight increase in the joint

strength was observed for both the systems. It has already been demonstrated that the hot shear strength improved as a result of the relief of internal stress in glass (Fig. 5). Thus we believe the aging treatment had a similar effect on the overall joining strengths. Compared with the 250°C shear strength data shown in Fig. 5, the hot-shear strengths of both $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ and AlN/AlN have improved after the aging treatment. It was already stated that the coefficient of thermal expansion (CTE) of low T_s glass frit and high T_s glass frit were $9.0 \times 10^{-6}/^\circ\text{C}$ and $7.8 \times 10^{-6}/^\circ\text{C}$, respectively. The coefficient of thermal expansion (CTE) of Al_2O_3 and AlN were $8.0 \times 10^{-6}/^\circ\text{C}$ and $4.6 \times 10^{-6}/^\circ\text{C}$, respectively. The stress formation due to CTE mismatch would be highest in the AlN/glass joint. The steady increase of the shear strengths of the AlN/AlN system (Fig. 6b) with aging time indicates that the aging treatment have a more significant effect to alleviate the stress caused by the CTE mismatch between glass and AlN [18, 21]. As a whole, the results reflect that the glass joint worked well as a solder material at higher temperatures ($\approx 250^\circ\text{C}$) in both ceramic systems.

Detailed cross-sectional studies were carried out to investigate the relationship between the shear strength and the interfacial morphologies of the double phase glass frit joints. Fig. 7 shows the SEM images of the interfaces between the ceramic substrates and the glass frit for all conditions. No significant changes were observed in the samples after 500 hours of aging. Overall the glass wetted both type of the ceramic surfaces very well and all the grooves on the ceramic surfaces were filled by the glass. No reaction product between the glass and the ceramic surfaces was observed within the resolution limit of the SEM.

The softening temperature (T_s) of one of the component is around 350°C , and that of the other component is around 650°C (Table 1). Therefore it is understandable that during bonding at peak

temperature (e.g. 420-440°C) the glass composition with high T_s would not melt, only the glass composition with low T_s would melt and complete the bonding action with liquid phase sintering. The two glassy phases are clearly distinguishable in the SEM micrographs as shown Fig. 8. The dark phases are distributed more uniformly in the white glass matrix. It should be noted that the high temperature melting glass powder loading in the low temperature melting glass matrix was 15 wt. %. Considering the chemical composition, Bi_2O_3 was the glass network former, while ZnO acted as the network modifier for the light grey phase. For the dark phase, both Bi_2O_3 and SiO_2 might act as network former, while the oxides of Mg and Zn act as network modifier.

Typical fracture surfaces on the substrate side are shown in Fig. 9 (a) and (b) of $\text{Al}_2\text{O}_3/\text{glass}/\text{Al}_2\text{O}_3$ and $\text{AlN}/\text{glass}/\text{AlN}$ joints, respectively aged for 100 hours. For $\text{Al}_2\text{O}_3/\text{glass}/\text{Al}_2\text{O}_3$ systems, the fracture surfaces were rough for both aged and non-aged samples. In these cases, the fracture passed through the glass matrix near the interfaces between the small substrate and glass matrix. This suggests that the interface between glass/ Al_2O_3 systems was stronger than bulk glass matrix within an $\text{Al}_2\text{O}_3/\text{glass}/\text{Al}_2\text{O}_3$ joint. Wave-like marks were noticed on the fracture surface for $\text{Al}_2\text{O}_3/\text{glass}/\text{Al}_2\text{O}_3$ joints (Fig. 9a). The high-melting glass particles might deviate the fracture direction during fracture propagation. It is expected that the high melting glass particles strengthened the bulk glass matrix through 2nd phase dispersion mechanism. For $\text{AlN}/\text{glass}/\text{AlN}$ systems, the fracture surfaces were relatively flat. In these cases, the fracture propagated through the interfaces between the small substrate and glass matrix for both aged and non-aged samples. This confirms that the interface between glass/ AlN systems was weaker than that of the glass/ Al_2O_3 systems. Even though no reaction product between the glass and ceramic surfaces was observed, the relationship of glass with Al_2O_3 substrates was found to be better than that with AlN substrates.

The voids in the bond structure had obviously significant impact on the reliability of the joints. Moreover the voids in the glass/ceramic interface would deteriorate the bonding strength drastically. The effects of the voids were not fully understood for the glass/ceramic joints as the voids in the glass phase were not measured quantitatively in this study. In general, the voids inside the glass joints need to be reduced to further increase the reliability of the ceramic/glass/ceramic systems. In this case, a little pressure is recommended to be applied onto the joints during bonding. This might squeeze out some of the voids from the glass melts at high temperature. From the μ CT and microstructural examination it was expected that the glass joint would be impervious to air or gases. Due to physical constraint, the designed package was not developed in the current study to check the actual leakage of the glass frits joint.

Conclusions

Based on the present findings, it can be concluded that the glass frit bonding could fulfill the requirement specified by oil and gas exploration industry for high temperature applications. It is found that the interfaces between glass/ Al_2O_3 systems were stronger than that between the glass/ AlN systems. However no reaction product between the glass and the ceramic surfaces was observed for both the systems. Among the two ceramic substrates, Al_2O_3 has been identified as a better candidate for the glass frit joining. Most importantly, the glass joints in both the Al_2O_3 /glass/ Al_2O_3 and AlN /glass/ AlN systems maintained their integrity even when shear-tested at 250°C . At higher temperature aging at 300°C for up to 500 hours, joint shear strength did not deteriorate. It is recommended that the two phase Bi-based glass frit mixture can be used as a joining material for harsh environment electronic applications.

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Table I The chemical composition of the glass powders.

Material	Glass powder 1 (Low Ts glass frit)	Glass powder 2 (High Ts glass frit)
Chemical Composition (wt.%)	Bi_2O_3 75-85 ZnO 10-15 B_2O_3 1-2	Bi_2O_3 30-40 SiO_2 25-35 Al_2O_3 17-25 MgO 6-10 ZnO 2-4 B_2O_3 2-4
Softening Temperature, Ts (°C)	350	650

Fig. 1 Ruggedized electronic packages employing glass frit as a joining material between the two ceramic parts.

Fig. 2 Schematic of the temperature profile for bonding (not to scale).

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