Al-Ge Diffusion Bonding for Hermetic Sealing Application

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The high-temperature requirement of Al-Ge eutectic bonding stands as a major obstacle in the wider acceptance for the hermetic sealing application among the micro-electro mechanical systems (MEMS) packaging industry, in particular for temperature sensitive devices. It has been demonstrated that the reduction in bonding temperature is feasible without compromising the hermeticity. The change in the mode of bonding from eutectic to solid-state diffusion did not have a dramatic impact on the bonding quality. However, this resulted in substantial increase in the bonding time. Shear strength too deteriorated as a result of the decrease in the thickness of the reaction interface. However, the shear strength still complied with the Military Standards (MIL). It has been confirmed that the hermetic seal could still be achieved without any solidification occurring at the interface. This is feasible since the inter-diffusion co-efficients of Al in (Ge) phase and Ge in (Al) phase are closer and are comparable to diffusion between solid-solution phases of identical metals like Au-Au, Cu-Cu, Si-Si bonding, which are generally used for such hermetic sealing application. Appropriate stacking mechanism for Al/Ge diffusion bonding is identified to overcome the limitations with respect to surface topography.

Key words*:* Eutectic bonding, diffusion bonding, solid solution phases, **C-**SAM, hermeticity, shear strength

INTRODUCTION

It is essential to explore alternative methods to alleviate the requirements of high bonding temperatures in order to cater for low thermal budget and temperature sensitive chips such as micro-opto electromechanical systems (MOEMS), infrared sensors etc. In addition, low temperature bonding approaches are looked into the industry with great benefits to overcome issues such as performance degradation under high bonding temperature (> 300°C), serious wafer/chip warpage, bonding misalignment and compatibility with the back-end process conditions and materials. In general, high bonding temperature must be reduced to avoid quality degradation due to the thermal damage of temperature-sensitive devices. Therefore, lowering the temperature for wafer direct bonding is a key issue in various future device applications [1-3].

Al-Ge eutectic bonding process is carried out in the temperature regime of 425°C and 475°C. This is because the eutectic bonding temperature of the Al-Ge system is around 420°C [4-5]. This high temperature requirement in this complementary metal oxide semiconductor (CMOS) compatible bonding technology is one of the major bottlenecks for the wide scale implementation. This is because of the degradation of the device reliability at such high process temperatures. The impact is evident in heterogeneous integration consisting of different materials. Therefore, low bonding temperature is highly desirable.

Al-Ge bonding has several advantages, when compared to the transient liquid phase bonding (TLP). The former is CMOS compatible and cost effective [6-8]. However, the high bonding temperature requirement restricts the wider application of Al-Ge eutectic bonding. In this work, Al-Ge bonding has been demonstrated at temperatures lower than 400°C, i.e., below eutectic. It has been proven that Al-Ge bonding too can be executed in the temperature regime similar to TLP bonding involving Sn, which is in the range of 280°C - 300°C [9]. Thus, this reduction in bonding temperature achieved would enlarge the application domain of Al-Ge bonding. It has also been proven that the void free interface serving as hermetic seal could also be achieved by diffusion between two non-identical metals, where the reactant and the product phases involve solid solution phases only, resulting in similar reaction kinetics across the interface and as a result, uniform flux of atoms leading to no accumulation of vacancies on either sides of the interface. Otherwise, diffusion between identical metals like Au-Au or Cu-Cu or Si-Si is generally preferred for this hermetic sealing application [10].

One of the major disadvantages of Al-Ge diffusion bonding, when compared to TLP bonding is that the former cannot take surface topography. This is because in TLP bonding one of the bonding metals, which is a low melting interlayer, is in the molten state during bonding. Appropriate, Al/Ge stacking has been identified for diffusion bonding in order to handle surface topography wafers. The Al-Ge diffusion bonding strength is lower than the eutectic bonding strength. However, it still complies with the Military Standard MIL-STD-883 requirements. Significant increase in the bonding time for Al-Ge diffusion bonding is the major drawback for this bonding technique, which could be addressed by surface activation and surface planarization methods.

EXPERIMENTAL PROCEDURES

Eight inch wafers were used for studying the Al-Ge diffusion bonding process. The width of the sealing ring varied between 80 µm -100 µm. The sealing ring pattern was defined by lithography and wet etching processes. Different sealing ring glass masks with varying device population have been used for this evaluation. Larger test vehicle was used for hermeticity testing. The undercut during wet etching process was ensured < 10 µm. Al and Ge thin films were deposited using the Blazer (Oerlikon Balzers Coating AG., Balzers, Liechtenstein) pulse vapor deposition (PVD) system. The deposition rates of Al and Ge were around 2 Å/sec and 6 Å/sec respectively. Method of stacking Al and Ge used in this work for diffusion bonding purpose is discussed in later section. Two different approaches were evaluated. In the first approach, Ge thin film is directly stacked on the Si wafer. Al is not placed underneath Ge. In the second approach, Ge is sandwiched between Al thin films during bonding. This approach is generally preferred, since increasing the number of bonding interfaces enhances diffusion bonding. The thickness of the Ge varied between 0.4 µm to 0.5µm. The thickness of the Al placed beneath Ge is 0.2 µm. The thickness of the Al stacked on the substrate wafer is 0.5 µm.

The diffusion bonding temperature was fixed at 350°C and 300°C. The bonding time and bonding pressure were varied, in order to determine the optimum condition. The variation of the bonding time was between 30 min to 2 h. The bonding pressure was varied between 11-35 MPa. Prior to bonding, the bonding wafer pairs were subjected to Ar etch pre-treatment for 1 min in the Balzer PVD system. The purpose of this pre treatment is to remove the oxides, in specific the aluminum oxides. The wafer level bonding was performed in a vacuum environment using EVG

Gemini Bonding System (EV Group, St. Florian am Inn, Austria). In-situ formic acid clean was done in the bonder for 5 min prior to bonding with the flags separating the substrate and the cap wafers. Germanium oxides are completely removed by this formic acid treatment. However, formic acid is not aggressive enough to remove stable aluminum oxides. Ar etching is determined to be a better pre-treatment method for removing aluminum oxide. However, the impact of Ar etching on the bonding quality could be realized only if the wafer level bonding treatment is performed within 4 h after Ar etching. Constant ramp rate of 35°C was used, since ramp rates do not have a major impact for diffusion bonding between solid-solution phases.

Qualitative assessment of the bond quality of the full wafer was performed using Sonix HS 3000 (Sonix, Spring field, VA, USA) scanning acoustic microscopy in C mode (C-SAM) with a 230-MHz transducer and a spatial resolution of 10 µm to 20 µm. The overall wafer level bonding quality was assessed by mapping the area, where water managed to seep through during C-SAM analysis. The bonded wafers were subsequently diced, using the Disco dicing saw equipment (Disco Corporation, Tokyo, Japan) at a dicing speed of 5mm/sec. Reliability of the bonded joints was verified by subjecting them to thermal aging at 200°C for 500 h.

Dies from different locations were picked for sample preparation for cross-section analysis using, JEOL 7600 (JEOL, Tokyo, Japan) scanning electron microscopy (SEM). Quantitative assessment of the bonded joints was performed using helium leak rate testing according to MIL-STD-883. The bonded dies are placed in a high helium pressure bombing chamber for 4 h, following which the samples were transferred to the helium spectrometer to monitor the leakage of helium.

The mechanical properties of the bonded joints were evaluated, using die shear tests. These bonded joints suppose to serve as a hermetic seal. The shear tests were conducted with a Dage 4000 shear tester (Nordson Dage, Aylesbury, Bucks, UK), employing a load cell capable of recording forces up to 100 kg. 10 samples were evaluated for each bonding condition before and after being subjected to thermal aging. The dies are selected from all regions of the wafer so that the reported results are representative of the wafer. The qualification of the bond strength was done by the compliance to the minimum requirement specified by the MIL-STD-883. The shear strength of those samples, where the failure was not at the interface has not been included.

DESIGN OF METAL STACKS

Two different metal stacking approaches were evaluated in this work. In the first approach, Ge was placed by PVD technique directly on the substrate wafer, as illustrated in Fig. 1a. In the second approach, Al is placed underneath Ge (Fig. 1b), resulting in Ge being sandwiched during the bonding process. The advantage of the second approach is that the increase in the number of bonding interfaces. This will facilitate enhanced diffusion, especially grain boundary diffusion and lattice diffusion.

In the first approach, good bonding results could not be achieved. Through scan analysis confirmed that the water leaked through most of the dies, as illustrated in Fig. 2a. In through-scan images, dark region represents air indicating that only few regions are hermetically sealed. Optical images confirmed that most of the dies are only partially bonded, as illustrated in Fig. 3.

This is because Ge is a hard metal. It does not deform and planarize during thermo-compression bonding. Thus, the presence of Ge limits the applicability of Al-Ge bonding for high surface topography wafers. It was determined that the issue could not be fully resolved despite increasing the bonding pressure and bonding time (Fig. 2b). C-SAM analysis confirmed that the bonding yield was only 60% despite significant increase in the bonding time up to 2 h and bonding pressure up to 23 MPa.

In the second approach, Ge was stacked over thin buffer layer of Al, which in the range of 0.2 µm. Al is a soft and malleable metal. Topography variations could be absorbed by Al and as a result, higher bonding yield could be achieved. Thus, for all investigations in this work, the second method of stacking was preferred. The Al layer beneath Ge should be of fine texture, as illustrated in Fig. 4, in order to deform and absorb non-planarities under high-pressure. In order to obtain such fine texture the sputter process deposition parameters such as substrate temperature, pressure of the Ar sputtering gas, deposition time, direct-current (DC) sputtering power and glancing angle, needs to be fine-tuned.

BONDING PARAMETERS OPTIMIZATION

Hermetic sealing of MEMS devices require high quality void free bonds. Hence, the bonding parameters were optimized with the sole objective of ensuring void-free joints [4,11]. The bonding parameters were optimized at wafer level for Al-Ge diffusion bonding. It was determined that it is not feasible to use prevalent eutectic bonding conditions for diffusion bonding. All the wafers bonded at 350°C using a bonding pressure of 10-15 MPa for 30 min delaminated during C-SAM analysis. Only when a bonding pressure of 18-24 MPa is used, the wafers did not delaminate.

Unlike the eutectic solidification, diffusion bonding consumes time. It requires longer bonding time. Diffusion by grain boundaries and lattice diffusion are slower in pace, when compared to the eutectic solidification. Conventional eutectic bonding at 430°C requires only 10-30 minutes for wafer level bonding. However, it is not feasible to achieve diffusion bonding at 350°C within 30 minutes. It was determined that a minimum of one hour is required to achieve void-free wafer level hermetic bonding at 350°C. Moreover, if the bonding temperatures are decreased further extended bonding times are required then.

It was not possible to achieve hermetic bonding within one hour, when the temperature is further reduced down to 300°C. Only partial bonding could be achieved, as confirmed by C-SAM images (Fig. 5a). For the same 300°C bonding temperature, when the bonding time was extended to 90 minutes, substantial improvement in the bonding quality was observed. Only few regions remained unbonded, as confirmed by C-SAM analysis (Fig. 5b).

It was determined that the reduction in diffusion bonding temperature would result in the increase in the diffusion bonding time. This increase is bonding time could not be compensated by the increase in the bonding pressure. It was determined that there is a minimum bonding pressure requirement for diffusion bonding (18-24 MPa), which is higher than eutectic bonding (10-15 MPa). No direct correlation between the diffusion bonding temperature and diffusion bonding pressure was observed. The diffusion bonding pressure requirement at 350°C as well as at 300°C is still the same. However, the diffusion bonding time requirement at 350°C is 60 min, while at 300°C, it is 90 min

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Although, hermetic bonding is achieved at longer bonding times, it is not feasible for high-volume production. The wafer level bonder could be efficiently used by introducing post thermal aging in the post bonding processes. The process time of Al-Ge diffusion bonding could be reduced by surface activation assisted diffusion bonding like plasma treatment. The diffusion time also could be reduced by surface planarization. If the surface roughness of the mating surfaces is controlled within 2 nm by means of bit grinding or any other planarization methods, the diffusion time could be dramatically reduced. Surface roughness is a critical parameter that determines the bond quality and is closely associated with the bonding time.

All the above optimized bonding parameters applies only to 8 inch wafer only. The focus of this article has been limited to 8 inch wafer. This is because MEMS devices, which require hermetic sealing is predominantly being fabricated at present in 8 inch wafer only. In case of 12 inch wafer, the bonding time needs to be re-optimized due to larger surface area. No change in the bonding temperature and bonding pressure. Bonding force has to be re-adjusted to achieve the required bonding pressure.

For thin wafers, considering the high bonding force involved, it is suggested to perform wafer level bonding first, followed by wafer thinning. Wafer level bonding could be performed on thin wafers without the need of support wafer up to 400 µm. Further thinned down wafers need support wafer, which are generally permanent and whose interface should be able to withstand the diffusion bonding temperature without any delamination. Suggested fabrication process flow is to perform the Al-Ge wafer bonding first, in standard 8 inch wafer with full thickness in the range of 675 µm to 725 µm, followed by wafer thinning. No delamination of the bonded wafer is observed, when the thickness of the bonded wafer is reduced until 200 µm by back grinding.

MICROSTRUCTURE CHARECTERIZATION

Coupled growth of eutectic phases was not observed in the solidified microstructure of the Al-Ge eutectic bonded wafer at 430°C, as illustrated in Fig. 6a. The white (Ge) phase is dispersed in the gray (Al) matrix phase. (Al) and (Ge) phases exist as discrete phases with limited solubility. Al is absolutely insoluble in the (Ge) phase, while the solubility of Ge in (Al) phase is less than 5 Wt. %. No any intermetallic compound (IMC) was observed in the microstructure.

A major change in the microstructure was observed, when the bonding was executed at below 400°C. Unlike the eutectic bonding, no solidified (Al) and (Ge) phases were observed in the microstructure of the bonded joints. The thickness of the reaction zone for the diffusion bonded joint is smaller than the eutectic bonded joint. In theory, thickness of the diffused zone is temperature dependant. However, only a minor variation could be observed in the respective microstructure between the samples bonded at 350°C and 300°C, as illustrated in Fig. 6b and 6c.

Diffusion bonding between different species across the interface is generally not preferred for hermetic sealing application. This is because there is a high probability of formation of Kirkendall voids as a result of the differences in the diffusion kinetics. Kirkendall voids formation occur closer to the interface on the side of the species with slower inter diffusion coefficeient. However, no voids was observed in the Al-Ge joint subjected to diffusion bonding indicating that the reaction kinetics of Al in the (Ge) phase and Ge in the (Al) phase are closer.

No significant change in the microstructure was observed between the diffusion bonded samples subjected to thermal aging at 200°C for 500 h (Fig. 7) and the ones which were not (Fig. 6). This could be primarily attributed to the low thermal aging temperature resulting in insufficient activation energy. However, minimal growth in the interface for the diffusion bonded samples subjected to thermal aging was observed in the respective micrographs (Fig. 7). This was confirmed by quantitative measurements too.

QUALITATIVE HERMETICITY CHARACTERIZATION

C-SAM analysis is used for qualitative assessment of the bond quality. The primary advantage of C-SAM over cross-section analysis or helium leak rate testing is that it reveals bond quality information of the full wafer. Cross-section analysis or helium leak rate testing is limited to few dies. In C-SAM analysis, the bonded wafers are immersed in water. Any leakage or seepage of water into the dies could easily be detected during the C-SAM analysis due to the difference in density [11]. The gray regions in the C-SAM image represent water, indicating the leakage of water through the hermetic seal. Thus, the presence of voids in the interface could be interpreted by C-SAM images. However, the C-SAM cannot detect minute voids in the hermetic joint, which is typically less than 20 µm. The C-SAM images of the optimized bonding profile for eutectic bonding and diffusion bonding are illustrated in Fig. 8. No major leakage was detected for these optimized bonding conditions. Void free hermetic joints could be achieved for Al-Ge diffusion bonding, provided that the proper pretreatment is preformed prior to bonding. The Al wafers are subjected to Ar etching from 25 to 40 cycles to mitigate the growth of aluminum oxides. Germanium oxides are removed by in-situte formic acid cleaning in the bonder chamber. The impact of Ar etching could be realized only within 4 h prior to bonding. Hence, bonding should follow immediately after Ar etching.

Water seepage was detected in the C-SAM images of the diffusion bonded wafer. However, it does not reflect the bond quality since the seepage is outside the hermetic seal. Bond quality is deemed to be poor only if the presence of wafer is detected within the sealed die. Minor leakage in few dies was detected in the wafer level Al-Ge diffusion bonding at 350°C for 60 min using 23 MPa (Fig. 9). However, in a wafer level bonding, bonding yield > 95% is acceptable.

QUANTITATIVE HERMETICITY CHARACTERION

Helium leak rate testing is used for quantitative hermeticity characterization. A larger test vehicle is used for this purpose, since fine helium leak rate testing results are valid only for packages with a minimum volume of 0.05 cm^3. The hermeticity of the bond quality is qualified using the MIL-STD-883 for fine helium leak rate testing. The fine He leak check consists of pressurizing the sample with a high He pressure for a few hours in a bombing chamber and subsequent transfer to a He spectrometer for He leak rate measurement.

It is determined that all eutectic bonded joints complied with the Military standards (Table I). Based on the specific volume, the bonded joint is considered to be hermetic, according to MIL-STD-883, if the leakage rate is less than $5*10^{-8}$ atm cc/sec. The hermeticity of the eutectic bonded joints is better than the diffusion bonded joints. The number of diffusion bonded samples, which complied with the MIL requirement is high for samples diffusion bonded at 350°C, when compared to the ones bonded at 300°C. No change in hermeticity was observed for the eutectic bonded samples, when subjected to thermal aging at 200°C for 500 h. However, minor improvement in hermeticity was observed for diffusion bonded samples under both bonding conditions, when subjected to thermal aging. Thus, it was concluded that thermal aging had an impact on hermeticity only for diffusion bonded samples. This could be attributed to the continuity of diffusion kinetics across the interface, as the system has not yet reached equilibrium.

BONDING STRENGTH CHARACTERIZATION

One of the most important mechanical properties for evaluation of bonding quality is bonding strength or toughness. High bonding strength is always required for the packaging structure since mechanical support is one of the basic functions of the package. It is presumed that the shear strength is representative of the joint strength [12,13]. Die-shear testing was performed according to MIL-STD-883.The die shear strength is defined as the maximum force required to break the attached bond. It is expected that the mechanical shear strength will depend greatly on various aspects of the bond quality [11]. Ten dies were selected from different bonded regions of the wafer for each bonding condition for shear testing so that the results are representative. The results are illustrated in Fig. 10.

It is obvious from Fig. 10. that the shear strength of the eutectic bonded joints is higher than the diffusion bonded joints. This could be primarily attributed to two reasons. Firstly, the thickness of the interfacial reaction zone of the eutectic bonded joints is higher than diffusion bonded joints. During the eutectic reaction Al and Ge undergoes solidification and the resultant eutectic microstructure encompasses hard (Ge) phase dispersed in the matrix soft (Al) phase. Unlike the eutectic bonded joints, only partial Al and Ge involves in the diffusion reaction as a result of grain boundary diffusion and lattice diffusion. It was determined that unreacted Al and Ge always exist in the wafer level bonding subjected to diffusion bonding. Secondly, the eutectic bonded joints undergo solidification and the resultant microstructure is composite type micro structure. Unlike the eutectic bonded joints, diffusion bonded joints do not undergo solidification and hence, dispersed phase strengthening is not feasible. As a result, eutectic Al-Ge bonding resulting in composite microstructure encounters a brittle fracture, while diffusion bonded joint faces a moderate ductile fracture during shear testing.

Among the diffusion bonded joints, it is observed that the joints bonded at 350°C possess relatively higher strength than the joints bonded at 300°C. However, it could not be explained with the help of microstructure for this minor difference in shear strength between the two diffusion bonding conditions. The required minimum bonding strength has been calculated based on the bonding area with respect to the recommendations of MIL-STD-883. Although, the shear strength of the diffusion bonded joints are lower than the eutectic bonded joints, they still comply with the MIL-STD-883 (Fig. 10).

Shear testing was also done on samples, which were subjected to thermal aging at 200°C for 500 h. No major variation in the shear strength was observed in the Al-Ge eutectic samples before and after thermal aging. This could be primarily credited to the composite microstructure of the Al-Ge eutectic alloy resulting in a stable microstructure. However, slight increase in the shear strength was observed in the diffusion bonded samples, when subjected to thermal aging at 200°C (Fig. 11). This could be primarily attributed to the minimal interfacial growth during thermal aging.

CONCLUDING REMARKS

It has been confirmed that the Al-Ge hermetic bonding is feasible well below the eutectic bonding temperature (< 400°C) at the cost of bonding time. It has been demonstrated that the solidification at the interface is not essential for hermetic bonding, provided that the inter-diffusion co-efficient of the species across the interface is closer. The increase in bonding time could be minimized by introducing surface activation and surface planarization processes. The low bonding temperature process would be appealing to a wider range of MEMS devices in particular for temperature sensitive devices like bolometer, optical MEMS devices. Reliability of the diffusion bonded interface is verified by thermal aging at 200°C for 500 h. Al-Ge diffusion bonding can substitute Au-Sn or Cu-Sn TLP bonding and better bond quality can be achieved than the TLP bonded joints. Moreover, the absence of IMCs in the interface is an added advantage for Al-Ge diffusion bonding over TLP bonding, since they are inherent brittle in nature and have a tendency to generate structural defects.

Based on the findings of this work, Al-Ge diffusion bonding can replace eutectic bonding for applications involving low thermal budget dies, which cannot sustain high processing temperatures. However, eutectic bonding is still preferred for devices that can sustain high processing temperatures. This is primarily attributed to the shorter bonding time, larger interfacial zone and homogenous composition, as a result of eutectic solidification.

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FIGURE CAPTIONS

1. Material stack of Al-Ge diffusion bonded samples.

2. Through scan images of Al/Ge stacks without placing Ge beneath Al (a) short bonding time and medium pressure (b) long bonding time and high pressure.

3. Optical images depicting partially bonded sealing ring from different locations of the wafer.

4. SEM- secondary electron imaging (SEI) illustrating fine Al texture achieved as a result of fine-tuning sputtering deposition process parameters.

5. C-SAM images of diffusion bonded wafers at 300°C using 23 MPa for varying bonding times (a) 60 min (b) 90 min.

6. Microstructure of Al-Ge bonded joints subjected to different bonding conditions (a) eutectic bonded (b) diffusion bonded at 350°C (c) diffusion bonded at 300°C.

7. Diffusion bonded joints subjected to thermal aging at 200°C for 500 h (a) 350°C (b) 300°C

8. C-SAM images of Al-Ge bonded wafers subjected to different bonding conditions (a) eutectic bonding at 430°C for 30 min using 12 MPa (b) diffusion bonding at 350°C for 60 min using 23 MPa (c) diffusion bonding at 300°C for 90 min using 23 MPa.

9. C-SAM image of Al-Ge diffusion bonded wafer under optimized process conditions indicating leakage of water into very few dies.

10. Shear strength measurements of eutectic bonded and diffusion bonded joints.

11. Impact of thermal aging on the shear strength measurements of eutectic bonded and diffusion bonded joints.

TABLE CAPTION

1. Impact of thermal aging on hermeticity for eutectic bonded and diffusion bonded samples.