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Experimental characterization of the mechanical behavior of two solder alloys for high temperature power electronics applications

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A B S T R A C T

An experimental investigation of two potential candidate materials for the diamond die attachment is presented in this framework. These efforts are motivated by the need of developing a power electronic packaging for the diamond chip. The performance of the designed packaging relies particularly on the specific choice of the solder alloys for the die/substrate junction. To implement a high temperature junction, AuGe and AlSi eutectic alloys were chosen as die attachment and characterized experimentally. The choice of the AlSi alloy is motivated by its high melting temperature T_m (577 °C), its practical elaboration process and the restrictions of hazardous substances (RoHS) inter alia. The AuGe eutectic solder alloy has a melting temperature (356 °C) and it is investigated here for comparison purposes with AlSi. The paper presents experimental results such as SEM observations of failure facies which are obtained from mechanical shear as well as cyclic nano-indentation results for the mechanical hardening/softening evaluation under cyclic loading paths.

Keywords:

Power electronics
High temperature
Shear
Creep
Nano-indentation
Hardening

1. Introduction

Nowadays, high-temperature power electronics packaging designed for the power electronics systems are paramount components of the electrical inverters in the hybrid electrical vehicles as well as the aeronautical and military engines. Mainly, a power electronic packaging is subjected to a high thermomechanical and power cycling as well as external environmental conditions such as moisture and corrosion. At the end of the packaging lifetime, the failure happens very often due to the voids growth and microcracks propagation in the solder alloy. The last constitutes the major cause of the lifetime reduction. To take up the wide temperature ranges and the high current densities, a new high temperature packaging generation is currently investigated. In power electronic applications, diamond based semi-conductors appear to be the solution in order to widely increase the capabilities of the power electronic converters [1]. Diamond is known to have exceptional thermal, electrical and mechanical properties. In order to make use of the exceptional characteristics of the diamond and increase the packaging reliability at high temperatures, a good choice of the die attachment is primordial to improve the thermomechanical behavior of the electronic device. Many technologies have been proposed in the literature [4,5]. The first

technology is concerning solders with a high melting temperature. In spite of the availability of high temperature solders in power electronics, researches involving this kind of materials are scarce. The implementation cost of these alloys remains the major obstacle to their use. It should be also denoted that most solders which operate at temperatures higher than 500 °C are ternary alloys that may potentially cause the formation of unfavorable intermetallic phases in the interfaces. But this was not the rule. For example, studies were conducted on some ternary alloys such as the ternary systems AuAgGe and AgCuSb. It has been shown that for an AgCuSb system, the wettability, the temperature melting, the quality of the interface as well as the microstructure, are favorable for operating between 400 °C and 500 °C. This system is also compatible with a Ni/Au metallization deposited on an AlSiC substrate. No cracks or brittle intermetallic phases are formed [15]. However, Sb is an element classified as toxic although it is not affected by the RoHS and WEEE directives. Works are underway to test AuAgSi alloy with the same kind of metallization [11].

A second technology proposed to solve the problem of solder fatigue is to replace soldering by another technique compatible with the power module service conditions. This is achieved by replacing the junction chip/substrate and wire bonding connection by a low temperature sintering technique involving more resistant materials and thereby, minimizes the residual stresses produced at the elaboration process. Sintering consists on a powder form alloy which is subjected to a thermomechanical process (temperature

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Table 1
Die attachment techniques for high temperature electronic applications [3,4,7,8,10,12,17,19].

	T_m (°C)	CTE (ppm/K)	Young's modulus E (GPa)	Thermal conductivity λ (W/m K)	Elect. conductivity (10^7 S/m)
80Au20Sn [10]	280	16	68	58	63
88Au12Ge [12]	356	12	80	52	-
88Al12Si [7,19]	577	22-23	70	121	32
Au/In [4]	454	-	-	-	-
Au/Sn [11]	532	-	-	-	-
Ag/Cu [3]	780	-	-	-	-
Ag/In [17]	850	-	-	-	-
Ag nanopowder [8,12]	961	19	9	240	41

and pressure) to achieve cohesion and densification of the particles at a temperature below the melting temperature of the basic components. Sintering additives are used to promote the process. In the electronic field, the most commonly used material is the powder of silver nanoparticles [2]. In this case, the resistance to active cycling can be greatly increased and a lifetime improvement of the electronic component can be obtained [6]. The major drawback of this technique is the relatively high sintering pressure able to cause premature cracks in the brittle module components especially in the silicon chips. There is also a challenge in the fixation and the alignment of the components before the pressure application. This innovative technique is already applied in industrial products [9,18].

Another prospective for joining is the exploitation of the diffusion mechanisms to produce a high temperature resistant bond at low temperatures [5]. The concept consists on assembling two parts with a low temperature melting metal, then performing a heat treatment to allow diffusion of the metal in solid or liquid phase to the substrates. The resulting bond has a melting temper-

ature higher than the heat treatment temperature. Among the possible junctions obtained using this technique, we find the junctions formed by the Au/In multilayer (resistant binary alloy) [4], Ag/In and Ag/Sn (silver-rich solid phase), Cu/Sn (copper-rich solid phase) and especially Au/Sn (gold-rich solid phase). For example, some studies lead to the achievement of a high strength junction between the metallization NiSi₂/Cr/NiCr/Au of a silicon carbide semiconductor and an AuSn alloy after annealing for 2000 h at 350 °C [11]. In fact, when Sn diffuses in the Au metallization, its percentage in Au is reduced which increase the melting temperature of the junction [3,20]. Particular attention was also devoted to the AuIn alloy with a highly adhering indium component having a melting temperature of 157 °C. The indium reflow is carried out at 200 °C followed by an annealing step at 400 °C to obtain an AuIn junction able to withstand temperatures up to 450 °C [4]. The economic constraints depending on the alloys cost and the elaboration process of the joints promote the use of indium with other metals such as copper or silver. In the case of Ag metallization, the procedure for obtaining a high temperature resistant junction with Ag

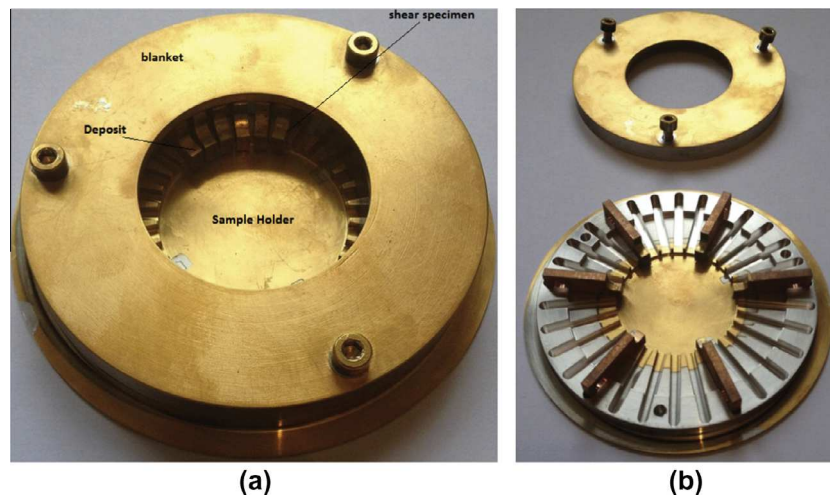


Fig. 1. Designed deposition tool for sputtering deposition. (a) Assembled and (b) decomposed.



Fig. 2. Designed high temperature titanium tool for the reflow operations. (a) Assembled and (b) decomposed.

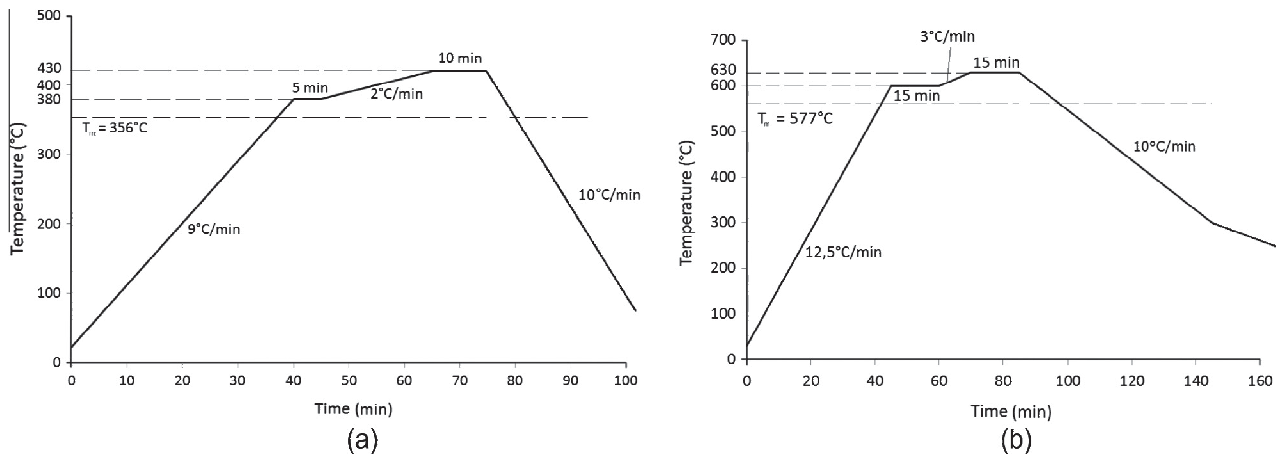


Fig. 3. Reflow profiles for (a) AuGe and (b) AlSi solder alloys.

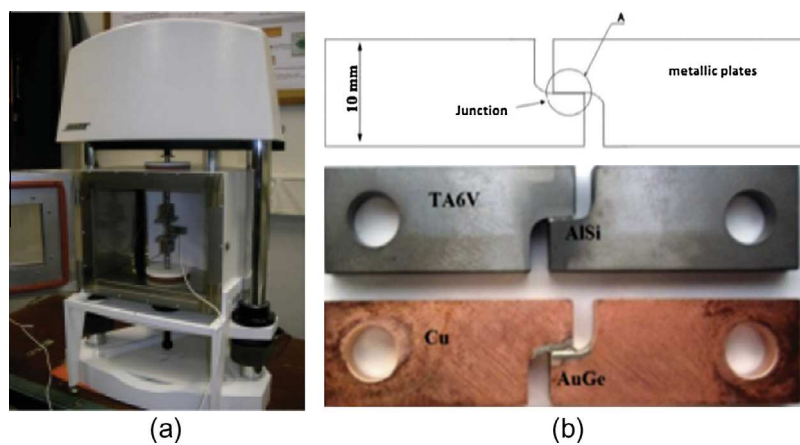


Fig. 4. (a) Fatigue testing machine used for macrostructural tests and (b) geometry of the used specimens.

and In is not unique. The junction can be obtained by either the deposition of an indium layer on a thick silver metallization or reflowing thin indium preforms just placed on the silver substrate. Using the first technique, a junction formed of a silver-rich solid solution was performed between a silicon based semiconductor with a Cr/Au/Ag metallization and a copper substrate coated Ag/In/Ag. The development process of the junction is performed at about 280 °C. After this operation, a very strong joint is produced formed by a stack of layers Ag/AgIn₂/Ag. Subsequently, attempts have been made to increase the diffusion of the indium layer and convert the AgIn₂ in a Ag layer without intermetallic phases [14]. The conditions for the production of a high-temperature junction such as temperature, annealing time and required thickness of the silver and indium layers, vary with the research frameworks but in all cases, the junction formed in the rich-silver solid solution can withstand up to 850 °C. Quintero frameworks permit to determine the influence of the experimental conditions on the quality of the obtained junction [17]. Another solution is based on the reflow of an SnAg alloy in order to diffuse Sn in the Ag metallizations. This technique is similar to the diffusion in the solid phase of AgIn but sets Sn instead of In. The same kind of junction is obtained with Sn using a silicon semiconductor coated Cr/Au and a copper substrate coated Ag/Sn/Au [21]. The thermal and mechanical properties of some relevant solutions for high temperature die attachment are summarized in Table 1 depending on the joining technology.

In this framework, two solder alloys for high temperature power electronic applications are investigated. The two joining

solutions are a high-temperature gold–germanium alloy 88Au12Ge and an aluminium–silicon alloy 88Al12Si. These eutectic solders have a high ductility and a good wettability on copper substrates. Their eutectic nature allows them to operate at high temperatures close to their melting temperature [3,12]. They have also high Young's modulus close to that of Aluminium and good thermal conductivity as shown in Table 1. Some limitations have to be verified for AuGe which is expected to exhibit creep effects at heating periods and residual stresses at the cooling stages. Monotonic and cyclic mechanical tests are primordial to prove or not the alloy thermomechanical capability. AlSi is a eutectic binary alloy, lead and gold free and then chosen for economical aspect. It is also compatible with the chosen thin metallizations for the high temperature packaging configuration. The AuGe solder is intended to be used with Ti/Cr/Au metallization scheme on diamond chip and Ni/Au for the copper bounded ceramic substrate. Whereas, Si/Al metallization scheme for the diamond chip and W-Si deposit for the DBC are fixed for the use with the AlSi solder. Parallel researches are ongoing in order to evaluate the mechanical behavior of these metallizations [6,13]. To explore the two die attachment solutions, some complementary tests have to be realized in order to determine the solder resistance to the mechanical and thermomechanical cycling. Among these tests, tensile, shear, creep and low cycle fatigue tests are commonly used for the prediction of the deformation behavior and the lifetime at various temperatures and strain rates. For these tests, a particular attention was made to the design of a single lap shear specimen. Afterward, SEM

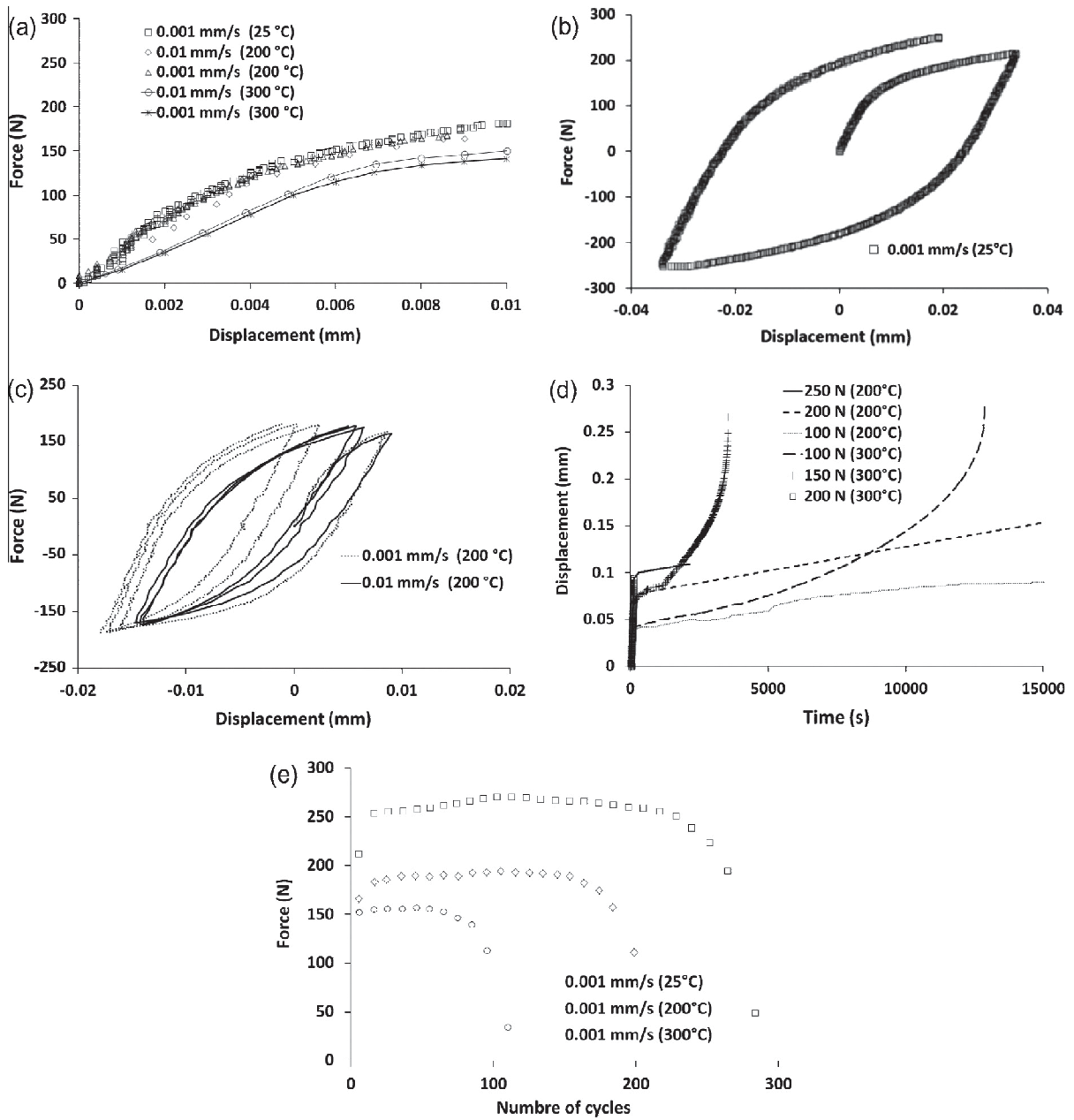


Fig. 5. Results of realized mechanical tests using AuGe shear specimen, (a) simple shear, (b) cyclic shear (1 cycle), (c) cyclic shear (5 cycle), (d) creep and (e) low cycle fatigue.

experimental observations of the failure facies of the samples are performed for the deformation and the present defects in the solder layers. Nano-indentation tests are also conducted to analyze the hardening and creeping behavior of the materials. Experiments at the nanoscale level such as nano-indentation and nanoscratching, are suitable for the materials characterization since they can be nondestructive and do not consume much material specimens. They are also reproducible, precise and do not require cumbersome efforts in the results treatments.

2. Experimental results and analysis

2.1. Macrostructural tests

At high temperatures, some solder materials exhibit viscoplastic mechanical behavior. In order to identify its dependency on temperature and strain rates for the two solders, a set of experi-

mental results, such as simple shear, creep and cycling shear tests are performed at RT, 200 °C and 300 °C using a single lap joint specimens [3]. These specimens are composed of two small plates of 3 mm thickness brazed to each other with the solder alloy. For the characterization of AuGe solder, the plates are made of copper while they are made of a titanium alloy Ti6Al4V for AlSi solder testing. The choice of copper as a substrate material is motivated by the good wettability of AuGe on copper. Due to the various brittle phases which are possible to appear in the Cu–Al binary system, Ti6Al4V plates are finally used for the experiments of AlSi characterization thanks to affinity of titanium with Al and Si.

2.1.1. Preparation of the samples

The Ti6Al4V plates are coated on the surface to be soldered with a thin Al layer of 500 nm thickness obtained by sputtering process using a designed circular aluminum device. The copper plates are kept without coating. The deposition device takes the form of a

circular wafer of 100 mm of diameter and can hold 30 metallic specimens to be coated with the sputtering technique. As shown in Fig. 1, the equipment consists of a cover tight by screws on a grooved sample holder. The shear samples are placed in the slots.

Before reflow, a cleaning operation of the copper plates is performed which consists on a degreasing with an acetone solution followed by a plasma assisted cleaning. Then, the AuGe samples are obtained after a reflow in a high temperature oven which can handle temperatures up to 400 °C under hydrogen and nitrogen controlled atmosphere. Besides, The Ti6Al4V plates are subjected to a degreasing in a sodium chloride solution, rinsing in water and a pickling in the hydrochloric acid. After the cleaning operations, the samples are placed in a high temperature titanium device containing 30 slots and shown in Fig. 2. As shown in this figure, the titanium cover ensures an intimate contact between the preforms and the plates. It is also designed to permit the alignment of the parts during reflow processes and to be mounted in the furnace.

The samples are then subjected to the reflow process. The reflow process for the AlSi samples is performed in vacuum using a high temperature furnace type "LALLIPUT" with radiation heat transfer and controlled reflow temperature. The furnace can operate until 1350 °C. The reflow profiles used for the two alloys are illustrated in Fig. 3.

The junctions are realized using solder preforms of 3 mm × 2 mm × 0.2 mm in order to have a brazed section of 6 mm². Some samples obtained after elaboration are shown in Fig. 4(b). The thickness of the samples are observed and controlled by optical microscopy.

2.1.2. Mechanical characterization

After elaboration, the specimens are mounted into an Electroforce® fatigue testing machine equipped with a thermal enclosure for temperatures up to 300 °C (Fig. 4(a)). For creep tests, the applied forces vary between 100 and 250 N. The measured dis-

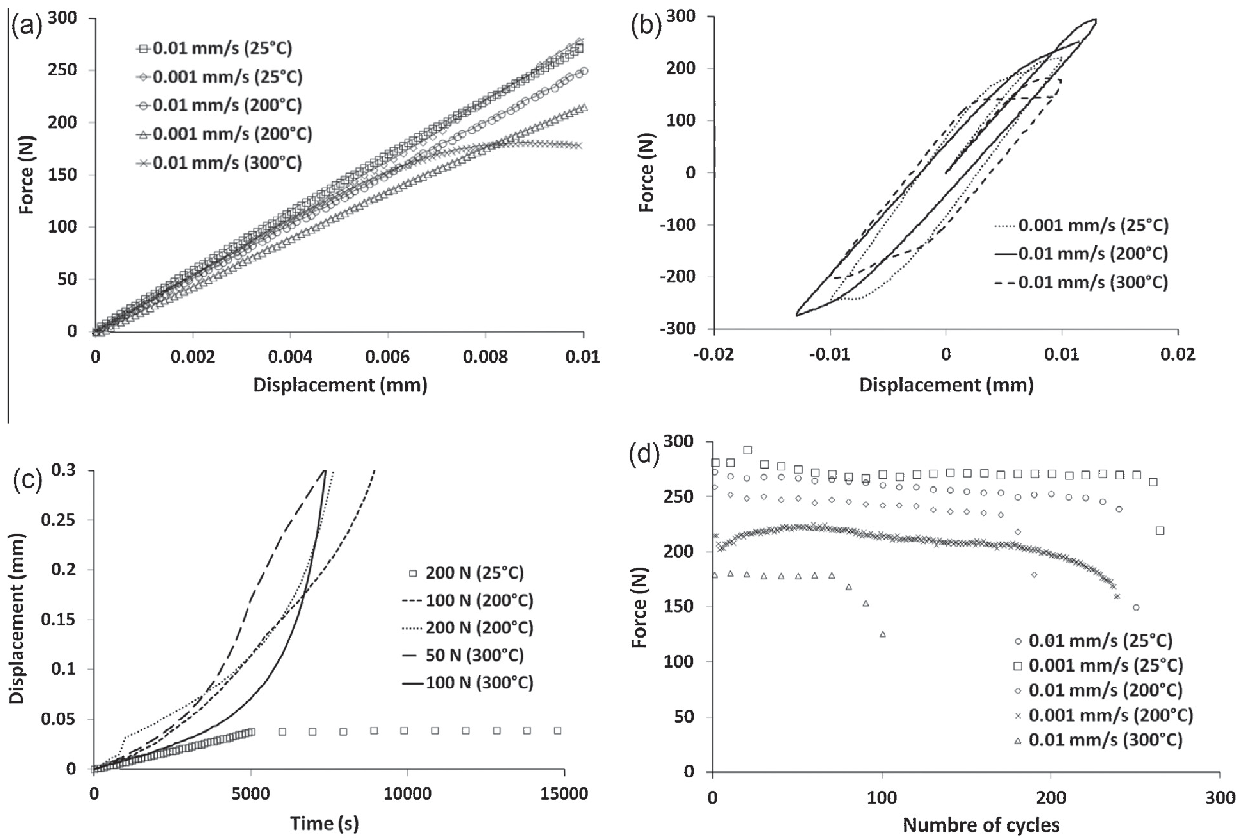


Fig. 6. Results of realized mechanical tests using AlSi shear specimen, (a) simple shear, (b) cyclic shear (1 cycle), (c) creep and (d) low cycle fatigue.

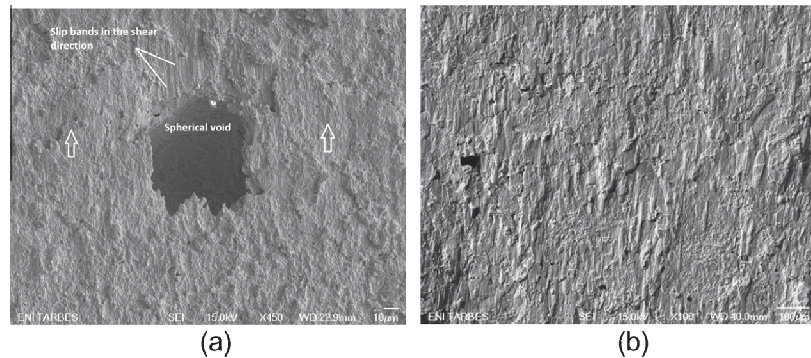


Fig. 7. Micrographs of specimen failure profiles. (a) Initial voids fraction in AuGe layer due to the reflow process and (b) shear bands in AlSi layer in the load direction.

placements are recorded using a miniature axial extensometer 3442 series from EPSILON Technology. The obtained samples and the fatigue testing machine are illustrated in Fig. 4.

In Fig. 5, some results of creep, simple and cyclic shear tests for AuGe and AlSi are presented. Fig. 5(a) shows the results of the monotonic shear tests made at different temperatures. AuGe alloy seems insensitive to the strain rate at temperatures less or equal to 200 °C. Thus, no viscous effects are observed below 200 °C. Indeed, between 25 °C and 200 °C, the tensile force does not vary so much regardless of the imposed displacement rate. For these two temperatures, the force reaches a maximum value close to 200 N for a displacement of 0.01 mm. At 300 °C, the maximum reached force decreases and takes a value of 150 N. It confirms that viscosity begins to be more consistent beyond 200 °C. The curves obtained from creep tests are shown in Fig. 5(d). No failure was observed for the samples tested at temperatures lower or equal to 200 °C for less than 4 h. The specimens tested at 200 °C remain in the stage of secondary creep (quasi-stable displacement) even after 15,000 s. However, the creep resistance decreases rapidly when the force increases at 300 °C. The failure happens after 220 min for 100 N and 70 min for 150 N. The displacement does not reach the tertiary creep regime for a force of 200 N. The cyclic shear test performed at 25 °C at an imposed displacement rate of 10^{-3} mm/s shows that the AuGe alloy undergoes a cyclic hardening in the first cycle (Fig. 5(b)). For a displacement of about 0.03 mm, the maximum load increases from 200 N to a value of about 250 N. This cyclic hardening continues until a temperature dependent threshold. Indeed at 25 °C, the produced hardening which is due to the increase of dislocation density has a preponderant effect and it is not negligible. Moreover, for force imposed cyclic shear tests, a displacement accommodation is observed over the first three cycles (Fig. 5(c)) which is due to the saturation of hardening. As in Fig. 5(e) in the case of a low cycle fatigue loading, the cyclic hardening stabilizes soon after the first few cycles of loading. The threshold is proportional to the test temperature. The maximal number of cycles to failure reached at 25 °C is about 300. The fact that the material hardens cyclically implies higher stress levels in the solder joint and increasing shear stresses on the interfaces. These high stresses are somehow transmitted in the packaging to the underlying components such as the die and the substrate.

Mechanical behavior of AlSi solder alloy is presented in Fig. 6. For the simple shear tests in Fig. 6(a), AlSi solder demonstrates a weak sensitivity to displacement rate even at high temperature. Moreover, it mainly has elastic behavior for temperatures until 200 °C and the applied force reaches a high value of 300 N. At 300 °C, the yield stress decreases and plasticity is reached (Fig. 6(a)). A fast saturation level is obtained due to the high material ductility and the increase of the dislocations annihilation mechanisms in the microstructural level. The AlSi mechanical behavior stills very close to that of aluminium. In the cyclic case illustrated in Fig. 6(b), there is also a weak amount of plasticity either for a temperature of 200 °C as shown in Fig. 6(b). Contrarily to AuGe solder, there is no Bauschinger effect (difference between tension and compression yield stress) for the AlSi solder but the material shows cyclic softening for the first cycle whatever the imposed displacement and temperature. This softening behavior is combined to the saturation of the force (200 N) at 300 °C. The creep curves shown in Fig. 6(c) do not permit to extract sufficient informations. The curves show no primary creep regime (first portion or high displacement rate part of the creep curve) except that at 25 °C in which the displacement stabilizes to a value of 0.035 mm. Time to creep rupture for temperatures of 200 °C and 300 °C does not change so much and is about 100 min. Finally, the low cycle fatigue tests performed at different temperatures and displacement rates (Fig. 6(d)) indicate that the numbers of cycles to failure are very close to those of AuGe at each tempera-

ture and that the cyclic softening of the material increases with the test temperature. This cyclic softening is benefic because it prevents higher interfacial stresses between the joined components which is suitable for the reliability of the power packaging.

2.2. Scanning electron microscope (SEM) observations

After specimen failure, observations at the microstructural level were carried out with a field emission gun scanning electron microscope (FEG SEM-7000F from JEOL with the incident electron beam maintained between 10 and 15 kV) to carefully examine the fracture surfaces or facies and shear bands of the broken specimens. Fig. 7(a) shows AuGe specimen failure facies after simple shear loading at 200 °C and a displacement rate of 6.10^{-2} mm/min. the morphology of the failure surface in this figure shows a striated structure. The failure profiles are heterogeneous but show that deformation hardening produces in the shear direction. However, the presence of an initial spherical voids fraction can be observed. These voids have an average diameter of 100 μ m. The presence of voids is essentially due to the solder reflow process during the samples elaboration and could be optimized to reduce their volumic fraction.

Fig. 7(b) illustrates the failure facies of the AlSi specimen. There are small shear bands oriented to the load direction. This layered structure characterizes a high plastic ductility of AlSi solder and proves that the failure occurs in the material volume. There is no apparent voids present in the solder layer after the reflow process and the mechanical testing of the solder.

2.3. Experimental results of nano-indentation tests

Single and cyclic nano-indentation tests are performed directly in the solder preforms using a MTS XP[®] nano-indentor with a Dynamic Contact Module (DCM) to determine the hardness and elastic properties [16]. Pay attention that the innovating aspect is the cyclic character of the test which comes from the fact that a single nano-indentation is repeated N times. It means that some time is allowed between each indentation at the same location. This cyclic loading profile permits to observe the accommodation

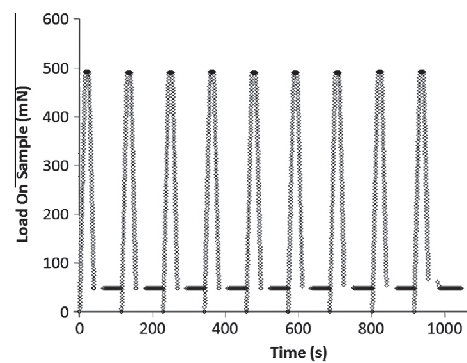


Fig. 8. Load profile applied for the nano-indentation tests.

Table 2
Nano-indentation test conditions.

Indentor type	XP
Tip geometry	Berkovich
Force resolution	100 nN
Column displacement resolution	0.1 nm
Number of nano-indentation cycles	10
Penetration depth	2 μ m
Approach rate	8 nm/s

of the displacement as it was illustrated later and thus show the cyclic hardening character of each solder. On each solder layer, 10 nano-indentation cycles and measurements in the Continuous Stiffness mode was carried out at RT with a Berkovich indenter by applying a maximum applied force of 500 mN. The cyclic profile of the nano-indentation force is illustrated in Fig. 8. It can be seen that the force is maintained for 10 s after the loading and the unloading steps.

In order to overcome the influence of the substrate and to take into account the surface roughness of the preforms, the interval for calculating the properties of the solders varies between 30 and 100 nm. The Young's modulus of the solders was calculated starting from the contact stiffness [16]. The whole set of the calibrated parameters of the nano-indentation device are summarized in Table 2.

The nano-indentation tests permits to measure hardness and Young modulus of both AuGe and AlSi solders. As shown in Fig. 9, the evolution of these two variables with respect to the tip displacement indicates that Young modulus saturates at about 80 GPa and 70 GPa for AuGe and AlSi respectively. Hardness stabilizes at about 2 GPa for AuGe and 1 GPa for AlSi. The dispersions recorded at small depths are mainly due to the biphasic nature of the alloys and the initial state of hardening induced by the polishing of the samples.

Following Fig. 10, the solder alloys AuGe and AlSi exhibit force vs. displacement curves completely different. The mechanical behavior differs radically between the first loading cycle and the remaining cycles. For the AuGe solder, the plastic deformation is very important during the first cycle ($W_{\text{plastique}} = 19 \text{ mN } \mu\text{m}$) and becomes very low in the second cycle ($W_{\text{plastique}} = 3.96 \text{ mN } \mu\text{m}$).

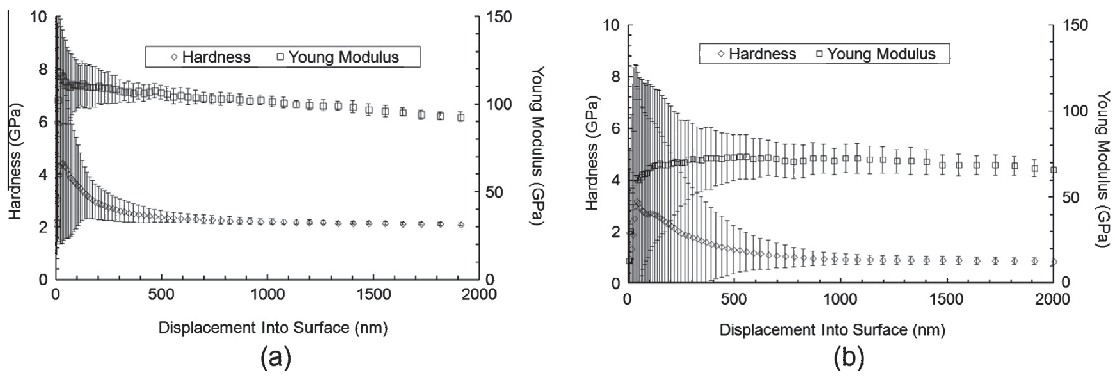


Fig. 9. Elastic moduli and hardnesses of the solder alloys (a) AuGe and (b) AlSi.

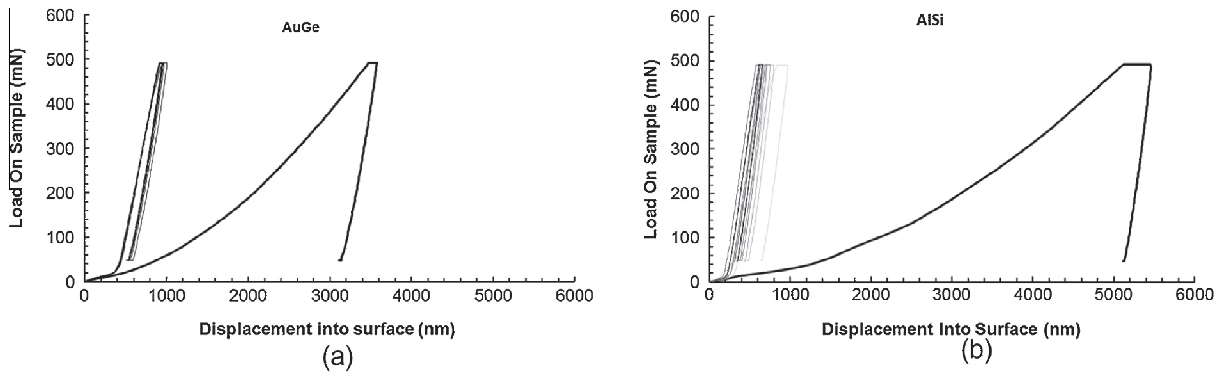


Fig. 10. Experimental curves of force vs displacement (a) AuGe and (b) AlSi.

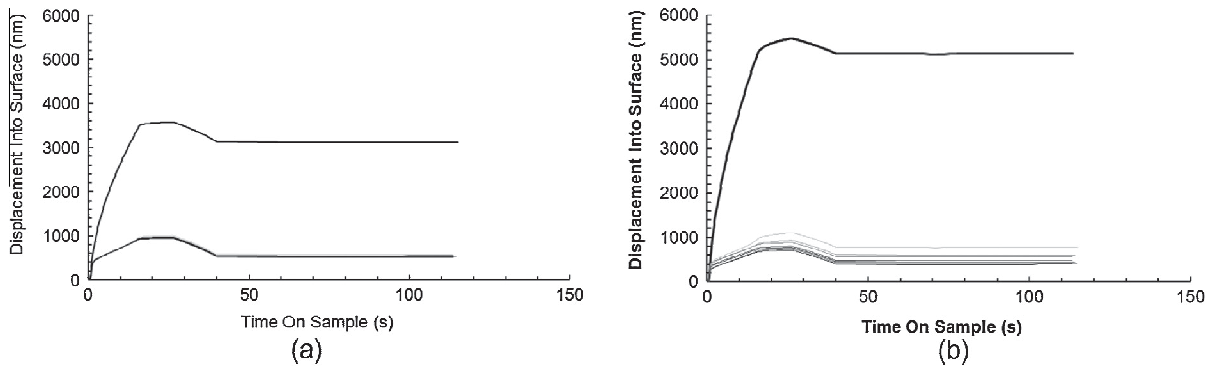


Fig. 11. Experimental curves of displacement with time (a) AuGe and (b) AlSi.

Due to little amount of kinematic hardening, AlSi solder alloy shows higher penetration distance. A higher creep deformation is also produced at periods of force holding. This is due to the static recovery mechanisms which act at constant loading conditions. This creep deformation decreases slowly until the fifth cycle of loading then stabilizes as shown in Fig. 10(b). However for AuGe, creep deformation is less observable and stabilization occurs faster. This can be also observable in Fig. 11 where displacement into surface is plotted as a function of time. For a given value of time, AlSi produces much more displacement than AuGe. In general, this hardening is comparable to ratchet phenomenon observed in the case of an asymmetrical positive load (case of cyclic mechanical uniaxial loading). Meanwhile, springback keeps the same value close to 100 nm for all the load cycles.

3. Conclusion

Uncoated copper substrates are joined using AuGe solder preforms. In spite of the possible formation of long-term intermetallic phases Cu_xAu_y due to the absence of a diffusion barrier in the short run, the creep and shear tests helped to solicitate the solder and characterize its thermomechanical behavior. The fractography shows the ductile deformation of the solder. Damage appeared around porosities. The use of the AlSi alloy for brazing Al coated TA6V plates is interesting with respect to the mechanical strength of the assembly. The obtained corresponding junction is very strong.

A study of AuGe and AlSi solder alloys was also conducted to determine their mechanical properties and the hardening behavior of each solder under monotonic and cyclic nano-indentation loads. Preliminary results show a cyclic hardening of both eutectic solders with a more pronounced hardening character for AlSi. AlSi appears to have an isotropic behavior very close to that of pure aluminum. The nano-indentation tests using monotonic and cyclic loads lead to the extraction of a set of informations concerning the mechanical properties of AuGe and AlSi. Mainly, the monotonic nano-indentation tests have permitted to find the Young's moduli of AuGe and AlSi ($E_{AuGe} = 80$ GPa, 71 GPa = E_{AlSi}) and to determine their hardness ($H_{AuGe} = 2$ GPa, $H_{AlSi} = 1$ GPa). The hardenable character which differs from an alloy to another is demonstrated by the cyclic tests. The profiles of the loading unloading curves show that for both alloys, there is a cyclic hardening. A higher rate of creep for AlSi is also recorded when the load reaches the maximum value. Finally, the cyclic tests present a remarkable point of divergence for both alloys. In fact, the displacement response for AuGe stabilizes after the third cycle. However, that of AlSi becomes stable after the 8th cycle. The AlSi solder appears to undergo cyclic hardening greater than AuGe solder.

Based on the low cycle fatigue results, AlSi and AuGe solders present very similar lifetimes but AlSi solder appears to be more appropriate for use in a diamond based power electronics packaging not only for its better mechanical properties especially the elastic stiffness but also its softening characteristic which allows it to "absorb" cyclically the stress level in the material. Thus, it allows less shear stresses in the components interfaces due to the CTE differences and avoids flexural failure of the die or the ceramic substrate due to the thermal cycling. AlSi presents also a little amount of defects based on the adopted reflow conditions which certainly plays a role in the fatigue lifetime of the solder. Subse-

quently, a focus on the mechanical behavior of the thin metallizations is also required for the temperature range of study in order to detect a possible formation of intermetallic phases between the metallization and the substrate. Moreover, the electronic performance of the diamond die especially the Schottky contacts under such high temperature must be fully understood especially when the diamond chip is subjected to the AlSi reflow conditions.

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