

An analytical review on interfacial reactions in high-temperature die-attach: the insights into the effect of surface metallization and filler materials

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Abstract. This review provides a comprehensive analysis of interfacial reactions and the impact of surface metallization in high-temperature die-attach, which is critical for ensuring the reliability of interconnects and joints in power electronic module packaging and integration. With the emergence of high-temperature filler materials, distinctive features in interfacial interactions and microstructural evolution arise, necessitating detailed examination to select suitable surface finishes based on the filler metals and specific applications. Metallization does not always enhance joint quality and reliability, so cost-effectiveness and manufacturability must also be considered when metallization is deemed viable. The formation of intermetallic compounds (IMCs) during interfacial reactions is particularly important, although solid solution formation at interfaces also warrants attention. This review evaluates five commonly used high-temperature metal solder fillers—high-Pb solder, Au-based solder, Bi-Ag solder, Zn-Al solder, and nano Ag paste—focusing on their interactions with various metallized surfaces in die-attach bonding. The effects of metallization on interfacial reactions and bond formation are discussed, leading to recommendations for cost-effective and reliable metallizations suitable for these applications.

Keywords: High-temperature electronics packaging / die-attach / metallization / interfacial reaction

1 Introduction

1.1 Recent progress in die-attach of wide-band gap (WBG) devices

With the fast-paced development of the electric vehicle (EV), aerospace exploration, and energy industry, electronic devices are now required to operate and perform in temperature ranges far higher than those of standard consumer electronics [1], as shown in Figure 1 [2]. To address this, wide bandgap (WBG) semiconductors have garnered increasing interest and are seen as a crucial advancement for next-generation electronics, offering a promising replacement for conventional Si devices [3]. The main advantages of WBG devices include higher breakdown voltage, faster switching speed and lower switching losses [4]. In theory, WBG semiconductors such as SiC and GaN can operate at high-temperature conditions up to 600 °C [1], as compared to the Si devices with the maximum operating temperature below 200 °C [5].

The rapid advancement of WBG devices and their applications places higher demands on die-attach joints to meet stringent mechanical, electrical, and thermal property requirements [6]. Table 1 presents the various characteristics of semiconductors at 300K as well as their maximum electronic operating temperatures [7]. Figure 2 presents the milestones of development of SiC power devices and their packages. Currently, packaging and integration are the primary bottlenecks limiting the full potential of these devices [8]. Conventional interconnection materials designed for less demanding conditions are generally incompatible with higher operating temperatures exceeding 250 °C. [9], particularly, in die-attach which is one of the most critical parts in first-level electronics packaging, which connects die and substrate [10].

Recent advancements in die-attach for WBG devices are largely driven by the development of advanced high-temperature interconnection materials and their potential manufacturing processes [11]. In addition to high-Pb solders, several other high-temperature solders have been introduced in recent years, including Au-based, Bi-Ag, and Zn-Al alloys. [12]. In addition, there has been a growing

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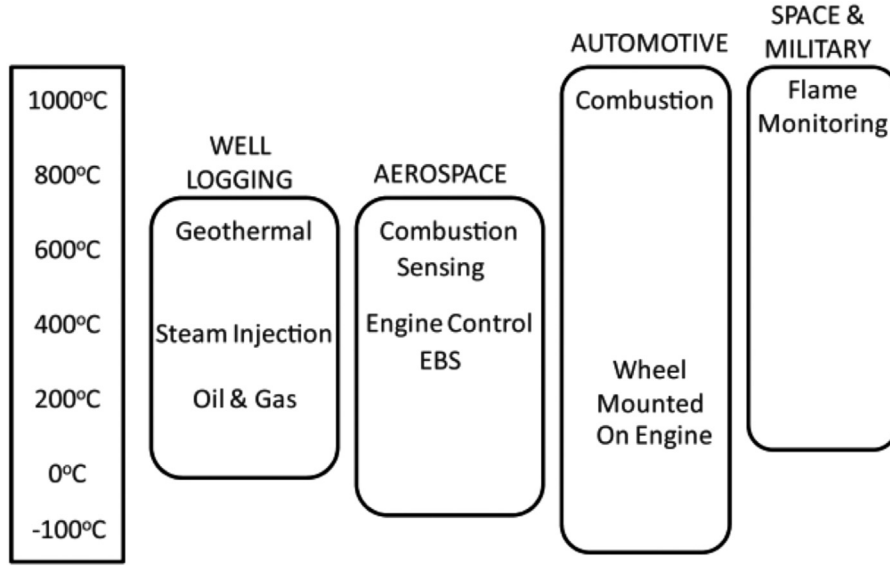


Fig. 1. Electronics devices in various critical industrial areas that need to be served under harsh environments [2].

Table 1. Properties of typical semiconductor devices.

Devices	Bandgap (eV)	Density (g/cm ³)	Thermal conductivity (W/cmK)	CTE (ppm/K)	Maximum operating temperature (°C)
Si	1.1	2.33	1.3	2.6	150
GaAs	1.43	5.32	0.55	5.73	350
3C-SiC	2.39	3.17	7	2.77	600
6H-SiC	3.02	3.21	7	5.12	700
GaN	3.44	6.1	1.1	5.4–7.2	>700
Diamond	5.48	3.52	6-20	0.8	1000

interest in metal paste sintering technologies, for instance, nano Ag sintering which has been utilised at various applications [13].

1.2 Bonding interfaces and failures in die-attach

The reliability of die-attach joints is closely linked to the quality of bonding interfaces, which are shaped by interfacial reactions during bonding. The primary failure mechanisms in die-attach structures include cracks, voids, and die/solder interface detachment. [14]. The cracks caused by thermal stress which are usually initiated at the die/solder interfaces resulting in 55% of the die-attach failure [15]. For joints formed by nano Ag sintering, the initial failure is commonly seen in the sintered Ag/die interface owing to the excessive thermal stress [16]. Thus, the interfacial reactions involved in joint formation play a crucial role in determining the failure mechanisms and lifespan of die-attach components during their operation.

Metallization applied to components is another critical factor that can significantly influence interfacial reactions, thereby affecting the formation of bonding interfaces and their microstructural evolution [17]. For example, it can serve as a barrier layer or enhance the wetting ability of

solders [18]. The substrate, whether it has metallized layers or not, interacts with filler materials (such as solders) to create bonded interfaces, which can be potential points of premature failure, particularly under high-temperature conditions [19]. By using different types of metallization, the interfacial reactions and the failure mechanism of resultant joints can be significantly influenced [20]. For instance, surface Ag finish on components (e.g. WBG dies) can significantly enhance the reliability of the attached die compared to Au finish when using nano Ag sintering under the same condition [21].

1.3 The scope of review

Despite the substantial research published in recent years on high-temperature die-attach materials, only a limited number have analyzed interfacial characteristics related to the applications and effects of metallization in conjunction with emerging die-attach filler materials. There is an urgent need to bridge this knowledge gap to better understand interfacial interactions in die-attach bonding, which will guide optimal metallization choices for specific filler materials and ensure high reliability. Initial motivations and efforts to develop various metallizations (such as

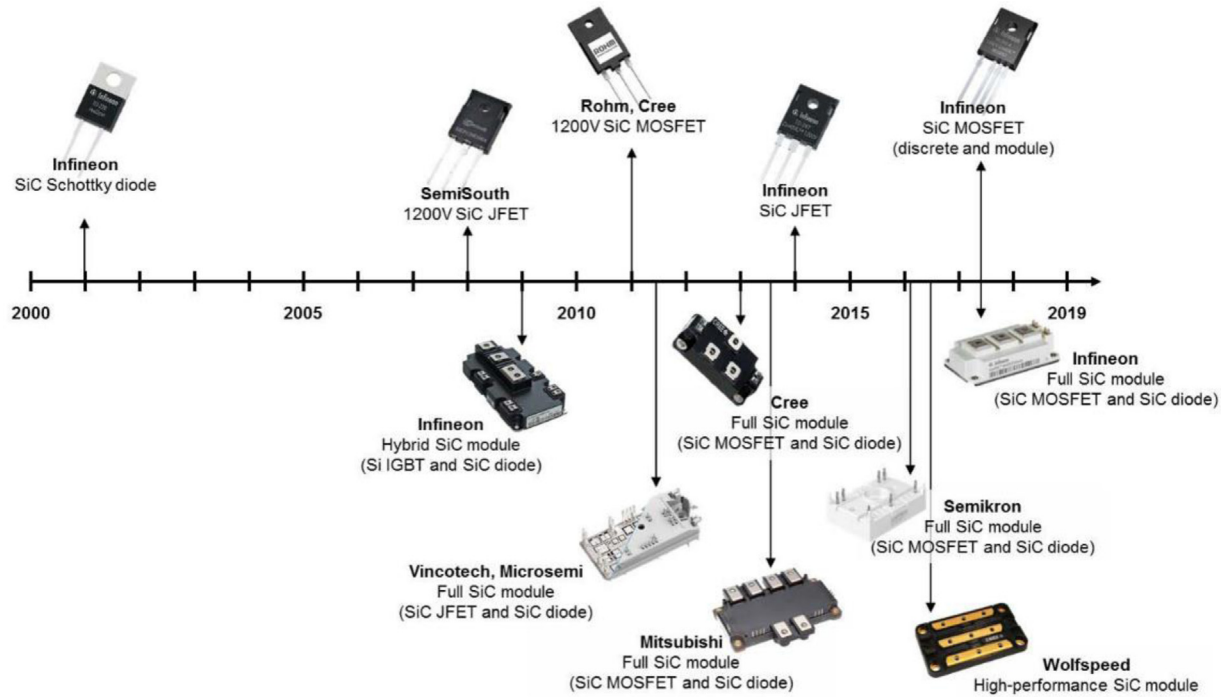


Fig. 2. Development of SiC devices and their packages [8].

Ni, Ag, and Au) were primarily aimed at addressing issues related to the use of high-Pb or Sn-based solders. However, the applications of metallizations have now expanded significantly with the introduction of new Pb-free filler materials, which are expected to be suitable for high-temperature die-attach in WBG devices.

Several reviews have addressed the properties of die-attach materials but have not highlighted the significant gap in the fundamental understanding of interfacial reactions during die-attach bonding processes, particularly concerning high-temperature applications and WBG devices. This current review paper examines and summarizes the applications and properties of several typical metallizations used on both die and substrate in the context of high-temperature die-attach applications. It includes a detailed analysis of the interfacial reactions involved in bonding processes with emerging high-temperature filler materials (melting points $\geq 250^\circ\text{C}$) relevant to the packaging or assembly of WBG devices. The paper compares and discusses the interfacial reactions, formation of bonded structures, and their performance based on various metallization and high-temperature filler combinations. Finally, it offers guidance and recommendations for the use of metallization across a wide range of filler materials to facilitate robust die-attach solutions for high-temperature WBG compound semiconductors in the future.

2 Metallization of semiconductor die and substrate

2.1 Die metallization

The backside metallization of a die is critical to enabling mechanical robustness, electrical contact, and thermal

conductivity for the interconnection materials. It usually contains multiple layers with each individual functions [19]. For instance, Ti/Ni/Ag is a classic metallization structure, in which Ti as the buffer layer to ensure the adhesion to the die and Ni as the diffusion barrier layer is able to prevent excessive interdiffusion during bonding process. The Ag as a surface finish layer can promote the interfacial bondability, for instance, with the interconnect filler metals such as nano-Ag or any solder alloys to enhance the interfacial adhesion and electrical conductivity [22]. In some studies, Au and Sn metal finish are also adopted to prevent the oxidation and sulfidation of the Ag layer [16,17]. Therefore, vapor-deposited Ag or Au are most likely to act as the terminal finish in various applications [24], e.g. Ti/Ni/Ag/Au multiple metallized coatings on Si die as typically observed and shown in Figure 3.

2.2 Substrate metallization

Typical substrates used in a power electronics module are usually a sandwich structure consisting of a thick ceramic sheet as main body for electrical insulation and the conductive layers (commonly Cu or Al) cold-fire bonded onto the both sides of the sheet to ensure the electric circuitry capable in the module [1]. Cu, as the most common upper conductor metal in a substrate, exhibits excellent electrical and thermal conductivity [25]. Cu also possesses desirable mechanical properties as an interconnection metal [26]. However, Cu is easily oxidized or corroded in a humid or oxygen-containing environment, hence a surface layer becomes necessary through surface modification for protection [27]. In addition, Cu can dissolve quickly in some lead-free molten solders and result

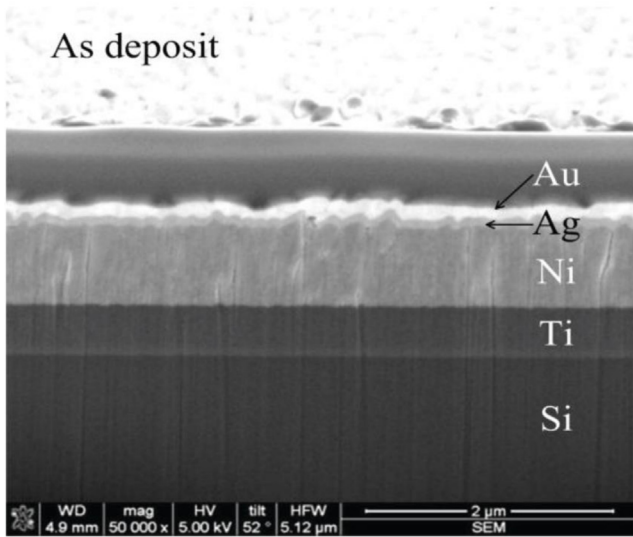


Fig. 3. Cross section of as-deposited Ti/Ni/Ag/Au metallization on Si die [23].

in an unstable microstructure, which can become a potential problem for reliability of the bonded structures [28].

Metallization is one of the most effective methods to eliminate the forementioned shortcomings of Cu substrate. The uses of metallization presents so many distinctive advantages with numerous choices of finish materials together with the tailored manufacturing routes. They may be considered and applied for a wide range of functional purposes usually including enhancement of solderability, prevention of the excessive dissolution of the substrate, and oxidation [29]. However, the cost is also an important factor when a metallization is applied, as well as associated interfacial reactions during the bonding, since cracks are likely initiated and propagate at the interfaces between the metallization and bare Cu substrate, causing the typical interfacial failure of the joints [30].

Ni coatings with a thickness of several micrometres are often used as a diffusion barrier layer to protect Cu substrate, since the reaction of Ni with molten solder is much slower than that of Cu [26]. The electroplated Ni coating on Cu has been applied in the electronics industry for many years, whilst the electroless Ni-plating method does not deposit pure Ni, but a mixture of Ni and P, which is brittle when P content is high (10–15%) and may result in failure [31]. Electroless Ni(P) coating can be conveniently deposited on the overall Cu surface with well-controlled composition [32] and the residual stress induced is much lower than the sputter and electroplated Ni thin coatings [33].

Ag and Au finishes have been used to protect surfaces from oxidation for enhancement of solderability, and they are chemically stable for a long storage time [34]. The most common coating structures and manufacturing methods include immersion silver (ImAg) [35] and electroless nickel/immersion gold (ENIG) [36]. Electroless nickel/immersion silver (ENImAg) has also been reported recently [37]. The other benefit of ENIG includes outstanding electrical

conductivity [26], excellent wear and corrosion resistance [31]. However, the high cost of Au limits the application of ENIG. In addition, ‘black pad’ is a known failure phenomenon due to the oxidation of electroless Ni(P) in excessive gold deposition area [38]. The general properties of various substrate and surface finish are listed in Table 2.

A schematic diagram of typical die-attach joint cross-section structure with metallization was presented in Figure 4, herewith two regions of interfacial reactions are involved in the die-attach bonding process, one is from the die-side with another from substrate-side.

3 Interfacial reactions between high-temperature solders and substrates

3.1 Introduction of high-temperature die-attach materials

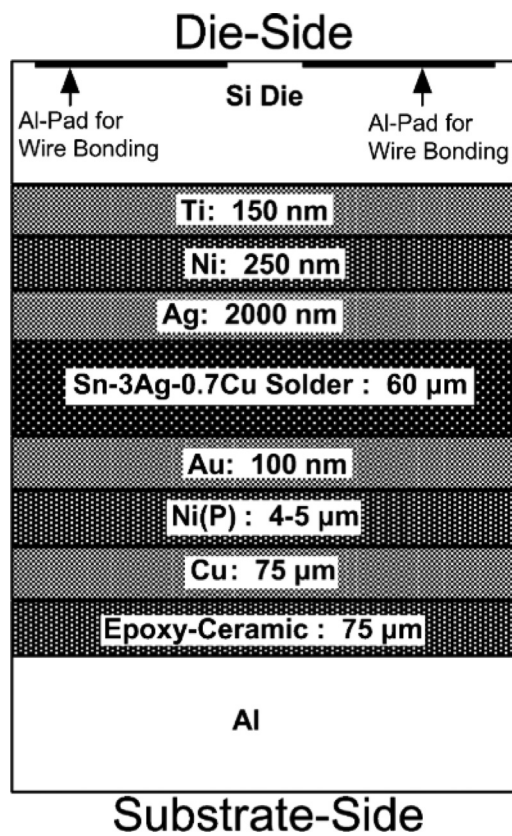
Significant research has been conducted in the literature to explore various aspects of high-temperature die-attach materials [40]. A die-attach material must have acceptable mechanical, thermal and electrical properties [9]. Additionally, the heat generated during device operation causes expansion throughout the entire module. Therefore, the coefficient of thermal expansion (CTE) of filler materials used for die-attach must account for the thermal mismatch resulting from the stress induced between the die and substrate [41]. In addition to the required functional properties mentioned above, it is essential for the materials to be cost-effective and manufacturable at low processing temperatures [41].

Currently, high-Pb (>85 Wt% Pb) solders are still the dominant materials utilised in high-temperature electronics packaging because of their suitable melting point (300–314°C) and proven reliability [42]. However, the high-Pb content in the solders has posed significant pressure on the legislation due to health and environmental concerns [43]. Several Pb-free candidates have been proposed, including Sn-Ag-Cu (SAC) system [44], Au-based [45], Bi-Ag [46], Zn-based [47] and nano metal paste materials [48]. The definition of ‘high-temperature’ can vary widely. For the current study, the high-temperature (HT) was defined as ambient temperatures above 250°C, which excluded the Sn-based solders currently used for consumer electronics.

The melting point is the primary criterion to consider for high-temperature filler materials. Table 3 presents the solidus and liquidus temperatures of both established and emerging interconnection material candidates. The selection of high-temperature filler materials is influenced not only by their melting points but also by their thermal, electrical, and mechanical properties, all of which are crucial for ensuring reliability in competitive die-attach systems. From this perspective, interfacial reactions play a vital role in forming die-attach bonds, which ultimately influence the performance and reliability of the entire assembled power module. Understanding the bonded microstructure and bond line interfaces resulting from these interfacial reactions is essential for grasping the fundamental effects of surface metallization and filler materials [49].

Table 2. Advantages and disadvantages of commonly used substrate and metallization in electronics packaging.

Substrate or surface finish	Advantages	Disadvantages
Cu substrate	Good mechanical property and thermal conductivity [26]	Easily oxidized or corrode, high solubility in molten solder [27,28]
Ni(P) metallization	Barrier layer	Cost effective, brittle if P is excessive [31]
Immersion Ag	Protect Cu from oxidation and corrosion [27]	High cost, micropores and defects in thin Ag layer [27]
Nickel Immersion Gold (ENIG)	Excellent solderability, excellent corrosion resistance [37]	Highest cost, use of cyanide, ‘black pad’ [37,38]

**Fig. 4.** Schematic diagram of a die-attach joint structure [39].

The general advantages and disadvantages of these die-attach materials are summarized in Table 4. It is essential to strike a balance between material properties, cost, and manufacturability when making a selection. Currently, there is no perfect filler material that meets all requirements among the existing candidates. It is critically important to associate the specific characteristics of these die-attach materials with the various substrate surface finishes or metallizations that are likely to be used for bonding. The pros and cons of these materials can be enhanced or mitigated to some extent through careful selection and application.

3.2 High-Pb solders

High-Pb alloys are currently the most widely used solders for high-temperature electronics packaging, particularly for the large area die-attach [59]. When exposed to a high temperatures environment, the microstructures of high-Pb solders can maintain stable due to their low modulus [60]. However, the use of Pb in these solders has been highly undesirable posing a great legislation pressure to be removed from industrial products in because of health and environmental concern [11]. For instance, the European Reduction of Hazardous Substances (RoHS) restricted the use of Pb in electronic and electrical equipment for consumer electronics [61].

However, Pb containing solders are currently exempted in automotive, defence and aerospace industry for electronics packaging and assembly because of their unique properties and no replacements in the foreseeable future. Firstly, the Pb in the alloy systems can reduce the wetting angle in the soldering process. For example, the wetting angle of molten eutectic Sn-Pb on Cu surface is $\sim 11^\circ$, while molten pure Sn on Cu surface is $\sim 35^\circ$ [33]. The typical high-Pb solders include Pb-5Sn and Pb-10Sn [57]. The concentration of Sn plays a critical role in the formation and retarded growth of interfacial IMCs [60].

Pb cannot form IMCs with bare Cu, it is Sn that reacted with Cu at the interface [62]. The cross-sectional images of the Cu/Pb-0.5Sn and Cu/Pb-5Sn interfaces are shown in Figure 5. The presence of Sn in Pb-Sn solders significantly enhances wettability on the Cu substrate by promoting the formation of Cu_3Sn at the solder/substrate interface. Figure 6 illustrates the microstructural evolution of the Pb-5Sn/Cu interface during aging at temperatures ranging from 175°C to 250°C over periods of 500–2000 h. It is observed that Cu_3Sn is the only IMC formed under all temperature and aging conditions, though its morphology varies depending on the specific aging conditions.

Similar to the reactions at Pb-Sn solder/Cu interface, interfacial reactions also occur between Pb-Sn solders and Ni [63], Ag [64] or Au [34] metallization. The types of Ni-Sn intermetallic compounds formed from high-Pb solders/Ni soldering reactions are also closely correlated to the Sn concentration [63]. When the Sn concentration was 5 wt.%, Ni_3Sn_4 formed first, and then Ni_3Sn_2 formed between the

Table 3. Solidus and liquidus temperature of typical high-temperature interconnection materials.

Interconnection material system	Composition (Wt.%)	Solidus temperature (°C)	Liquidus temperature (°C)
High-Pb	Pb-5Sn [50]	300	314
	Pb-2.5Ag [50]	304	304
	Pb-1.5Ag-1Sn [50]	309	309
Au Alloy	Au-20Sn [50]	280 (eutectic)	280
	Au-3.2Si [51]	363 (eutectic)	363
	Au-12Ge [51]	361 (eutectic)	361
Bi Alloy	Bi-2.5Ag [50]	263 (eutectic)	263
	Bi-11Ag [50]	263	360
Zn Alloy	Zn-5Al [52]	381 (eutectic)	381
	Zn-4Al-1Cu [53]	380	380
	Zn-4Al-3Mg-3Ga [54]	309	347
Nano paste	Ag [55]	961	961
	Cu [56]	1064	1064

Table 4. Advantages and disadvantages of alloy systems for high-temperature die-attach [57,58].

Materials	Advantages	Disadvantages
High Pb	Suitable melting temperature, good wettability and mechanical properties, stable microstructure	Toxic, sensitive to Sn concentration
Au-Sn	Fluxless, good creep resistance	Expensive, brittle Au-Sn IMCs
Au-Ge	Low elastic modulus, stable microstructure, high strength	Expensive, difficult to manufacture
Zn-Al	Low cost, easy to manufacture, no IMC formed	Highly corrosive, poor wetting ability
Bi-Ag	Acceptable melting point, affordable cost	Poor workability, low conductivity
Nano Ag	High electrical and thermal conductivities, low processing temperature	Expensive, need pressure during sintering

Ni_3Sn_4 and Ni [63]. The growth of IMC layers at the Pb-5Sn/Cu and Pb-5Sn/Ni interface with time under different temperatures is shown in Figure 7. It can be found that the thickness of Ni_3Sn_4 is much thinner than Cu_3Sn , which exhibited the diffusion barrier effect of Ni metallization [63]. As for electroless Ni-P coating, a P-rich layer can be formed because of the Ni consumption by Sn during IMCs formation, thus Kirkendall voids were resulted invariably at the IMC/Ni-P layer interface [31].

Ag_4Sn was found as the resultant IMCs of interfacial reactions at the Pb-5Sn/Ag interface. Figure 8 shows the

cross-sectional microstructures of the Pb-5Sn/Ag interface reacted at 350 °C for 5–30 min, coarsening of the Ag_4Sn phase was also observed with increase of reaction time [65]. It can be found that the growth of Ag_4Sn is even quicker than that of Ni_3Sn_4 on Ni metallization compared to the results in Figure 8. The work also revealed that Au can be quickly dissolved into the molten eutectic Sn-Pb solder [33], forming a brittle and needle-like AuSn_4 phase which also migrated to the Ni interface during thermal aging, leading to the formation of An (Au,Ni) $\text{Sn}_4/\text{Ni}_3\text{Sn}_4$ interface which exhibited severe brittleness.

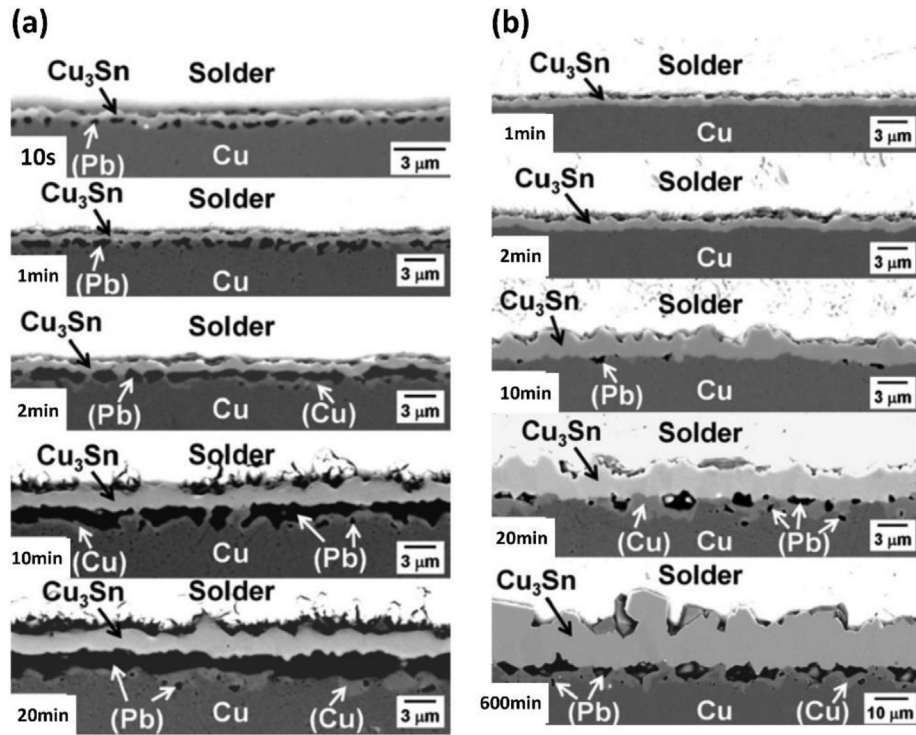


Fig. 5. Microstructure evolution for the reaction between (a) Pb-0.5Sn and (b) Pb-5Sn with Cu at 350 °C for different reaction times [62].

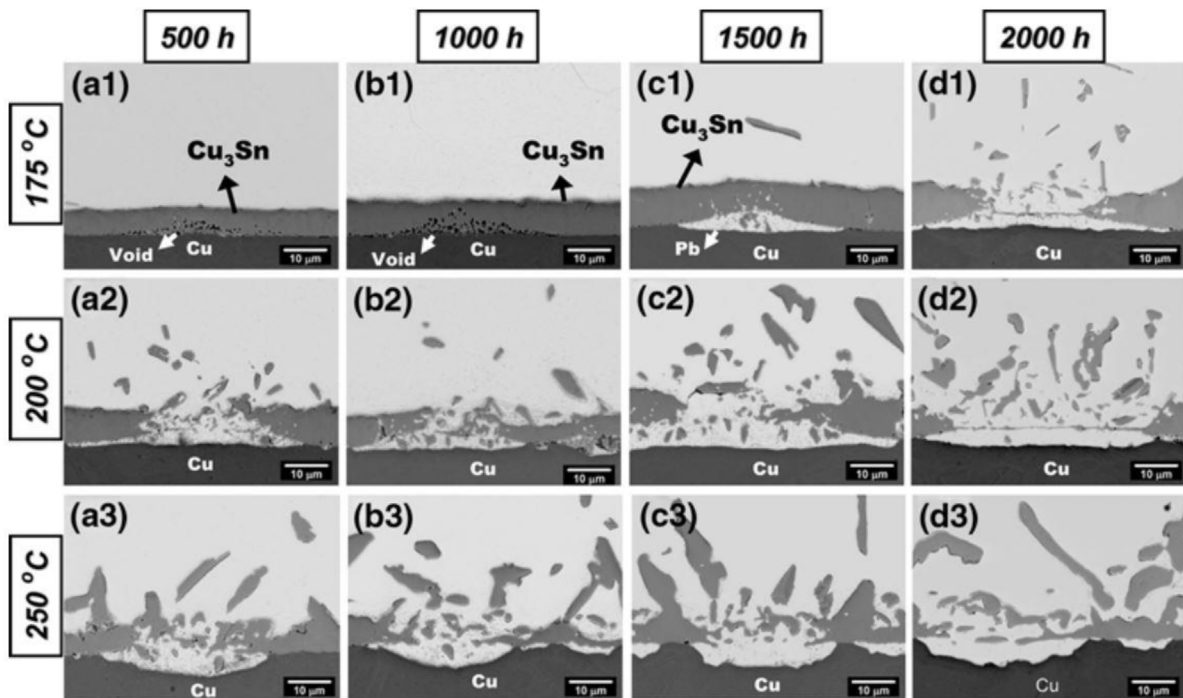


Fig. 6. Morphological evolution of Cu₃Sn at 95Pb-5Sn/Cu interface during aging [63].

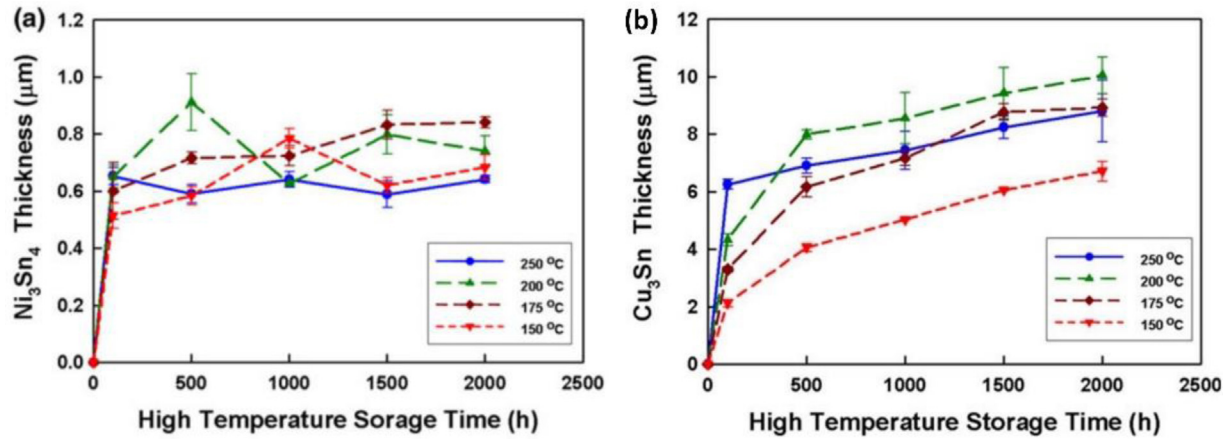


Fig. 7. Average thickness of intermetallic layer for Ni/95Pb-5Sn/Cu diffusion couples: (a) Ni_3Sn_4 on Ni side and (b) Cu_3Sn on Cu side [63].

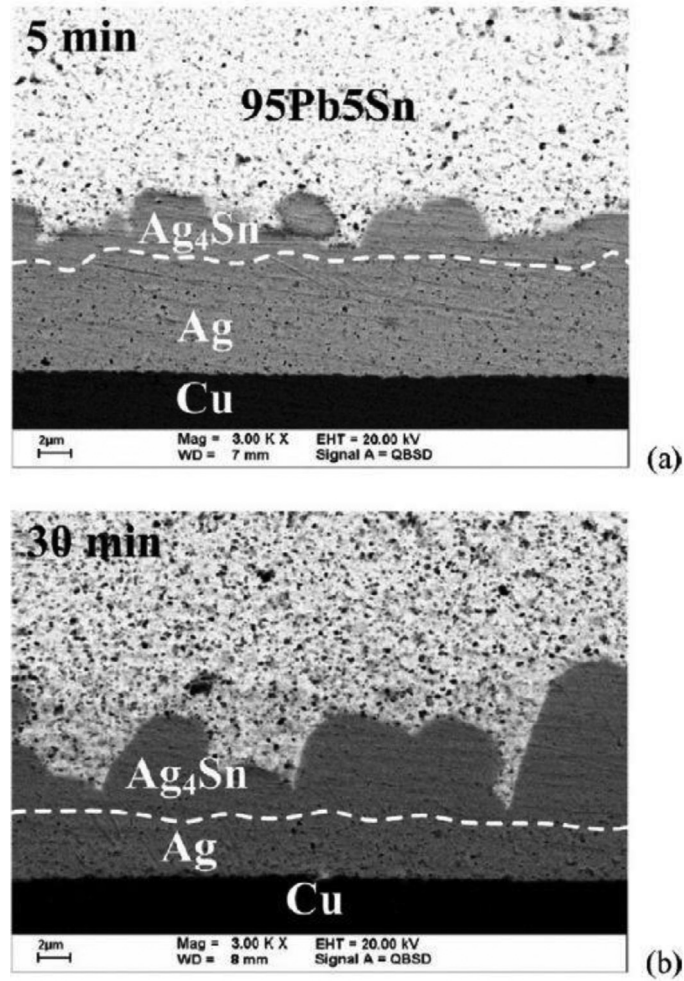


Fig. 8. SEM micrographs showing the cross-sectional microstructures of the 95Pb5Sn/Ag interfaces reacted at 350 °C for (a) 5min, (b) 30min [65].

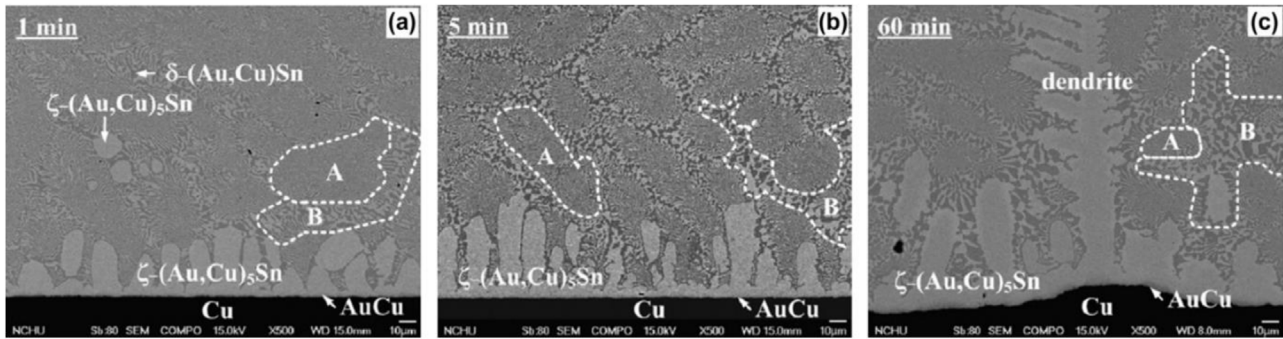


Fig. 9. Interfacial microstructure of the Au-20Sn/Cu solder joint after reflow for (a) 1 min, (b) 5 min, and (c) 60 min [75].

In summary, Pb-Sn solder can participate in the interfacial reactions with all types of substrates and metallization. It was the Sn element that is primarily involved causing the formation of IMCs subject to the surface finishes, however, Pb hardly reacted with any substrate metals, thus contributed to the stability of joint microstructure. Ni layer is effective as a diffusion barrier layer to reduce the reaction and growth rate of IMCs with Pb-Sn solder compared to Cu substrate. Ag and Au finish can still form IMCs with Sn at the interfaces. It is implied that the benefit of Ag and Au finish within this context is limited given the good wettability of high-Pb solders onto wide range metal finishes. The extra cost in the uses of Ag and Au metallization presents an obvious obstacle for the die-attach bonding with high-Pb solders.

3.3 Au based solders

Au-based solders have been widely used in high-temperature electronics packaging owing to their high thermal conductivity, mechanical properties and reliability [45,66]. In addition, they also have excellent wettability, which makes fluxless soldering possible [67]. Au-based solders are currently used for high-temperature electronic assemblies including microwave devices, laser diodes, and RF power amplifiers [59]. Among all the die-attach filler materials, cost factor presents a major hurdle to increase the volume of usage of Au-based although they are still considered for certain high-end applications due to their excellent properties, particularly where the cost may be justified and no alternatives available [68].

Au-Sn, Au-Si and Au-Ge alloys are the three most commonly-used Au-based solders with melting points below 400 °C, which are comparable with the high Pb solders [51,60]. The eutectic Au-20Sn alloy has a melting point of 280 °C, which is the most popular one among Au-based solders [69]. In such a system, two brittle IMCs phases can be formed inside the Au-20Sn joints, i.e. Au₅Sn and AuSn, and they are chemically stable and can enhance the strength of soldered joint [70]. The eutectic Au-Sn solder is found to have a longer thermal life than silver paste for high power LED packaging [68], which is highly desirable in the packaging of WBG devices.

Au-Ge alloy has high thermal and electrical conductivity, mechanical properties and corrosion resistance [71]. Like Pb-Sn and Zn-Al solders, no intermetallic phases may

be formed in the alloy system. The eutectic melting temperature of the Au-Ge (12 wt.% Ge) alloy is ~360 °C [66]. Moreover, it is unlikely for Ge to react with some common substrates and form any IMCs [45]. Au-Ge eutectic solder exhibits better wettability than Au-Si solder as Si is easily oxidized during soldering [32]. Au-3.2Si eutectic solder has a melting point of ~363 °C [72]. In comparison with Au-Ge joint the microstructure of the Au-Si joint is chemically less stable. The grain coarsening in Au-Si eutectic alloy is more pronounced when compared with the Au-Ge eutectic alloy [73].

On the bare Cu substrate, Au-20Sn can form a thick (Au,Cu)₅Sn IMC layer as well as a thin Au-Cu IMC layer [70]. This Au-Cu layer can grow quickly above 200 °C with the formation of a new Cu₃Au layer. A Ni metallization can effectively reduce the dissolution of the Cu substrate. However, the thin (Au,Ni)₃Sn₂ is also not stable during thermal aging, which can be transformed into (Au, Ni)Sn layer [74]. As for Au-12Ge and Au-3.2Si on the Cu surface, they are likely to have interfacial delamination within the IMC layer under 300 °C aging for 300 h. The wetting of liquid Au-12Ge solder alloy in contact with a Cu substrate reveals pronounced dissolution of Cu and fast diffusion of Cu into the liquid solder [71]. Chung et al. [75] investigated the reflow reactions of the Au-20Sn solder on a Cu substrate at 330 °C. Following a typical reflow time of 1 min, the solidified solder matrix exhibited a lamellar eutectic microstructure of (Au, Cu)₅Sn. As depicted in Figure 9, two distinct phases formed at the solder/Cu interface: irregular (Au, Cu)₅Sn and a layered AuCu structure. In this system, Cu behaves similarly to Au, leading to a shift in the solder composition from eutectic to hypo-eutectic as the Cu concentration increases.

It is not rare to use ENIG finish for bonding with Au-based solders, in such a case the cost of ENIG is no longer an issue compared to the expensive Au-based solders [45,73]. On an ENIG finish, Au-20Sn exhibits a similar interfacial reaction with Ni metallization, as shown in Figure 10. The entire Au layer on the Ni(P) layer rapidly dissolves into the molten solder during the reflow process [67]. Au-12Ge can react with Ni to form thin and stable Ge-Ni IMCs layers after long-term high-temperature operation [45]. On an ENIG surface, Ni₃Ge₅ or NiGe IMC can be formed with Au-12Ge, while AuNiSi is formed with Au-3.2Si. Finally, the interfacial failure for both

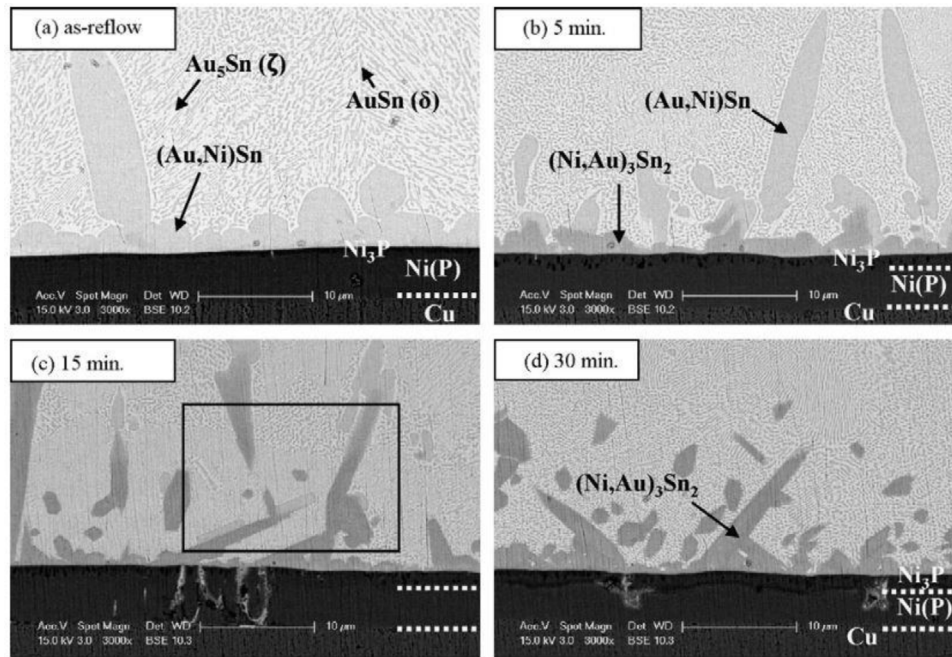


Fig. 10. Cross-sectional SEM images of Au-20Sn/ENIG interfaces reflowed at 310 °C for various times [67].

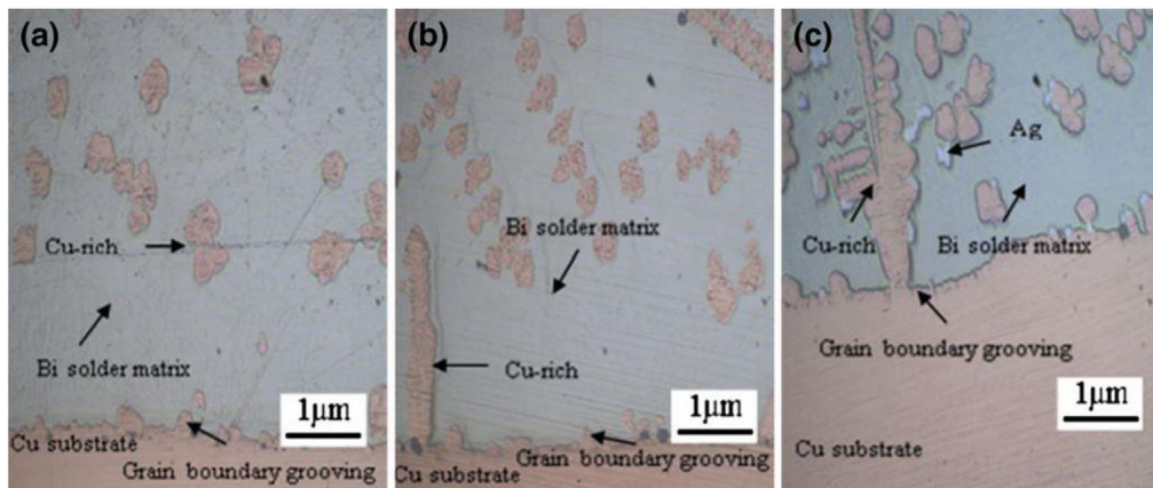


Fig. 11. Cross-sectional optical images for a sample Bi-1.5Ag, b sample Bi-2.5Ag, and c sample Bi-3.5Ag in Bi-Ag solder bulk [79].

Au-12Ge and Au-3.2Si on ENIG finishes is barely seen under the same aging condition compared to the Cu metallization [51].

To maintain the composition and microstructure of the Au-based solder joint, it can be concluded that Ni metallization is worthwhile to be applied as a diffusion barrier layer [45]. The cost of ENIG is acceptable when the Au-based solders are used. In addition, the Au finish can protect Ni from oxidation, which enables fluxless soldering.

3.4 Bi-Ag solders

Bi has a melting point of ~270 °C, which is a candidate for a die-attach material [7]. Bi-based alloys have several

drawbacks such as low electrical and thermal conductivity and brittleness [76]. The primary source of Bi is the by-product of Pb refining, which may limit its application [33]. The Ag additions were effective in strengthening Bi alloys. The Bi-2.5Ag eutectic solder exhibits an acceptable melting point (262.5 °C), which has an affordable cost and a similar hardness to that of Pb-5Sn [64]. Thus, it has been developed as die attach solders for power devices and light-emitting diodes (LEDs). Due to the brittleness of the Bi element, Bi-2.5Ag has relatively poor bonding strength and processability. When Ag content increased in the Bi alloys, the thermal conductivity improved [77]. When Ag content in Bi-Ag reaches to ~11 wt.%, the thermal conductivity can be improved significantly [59].

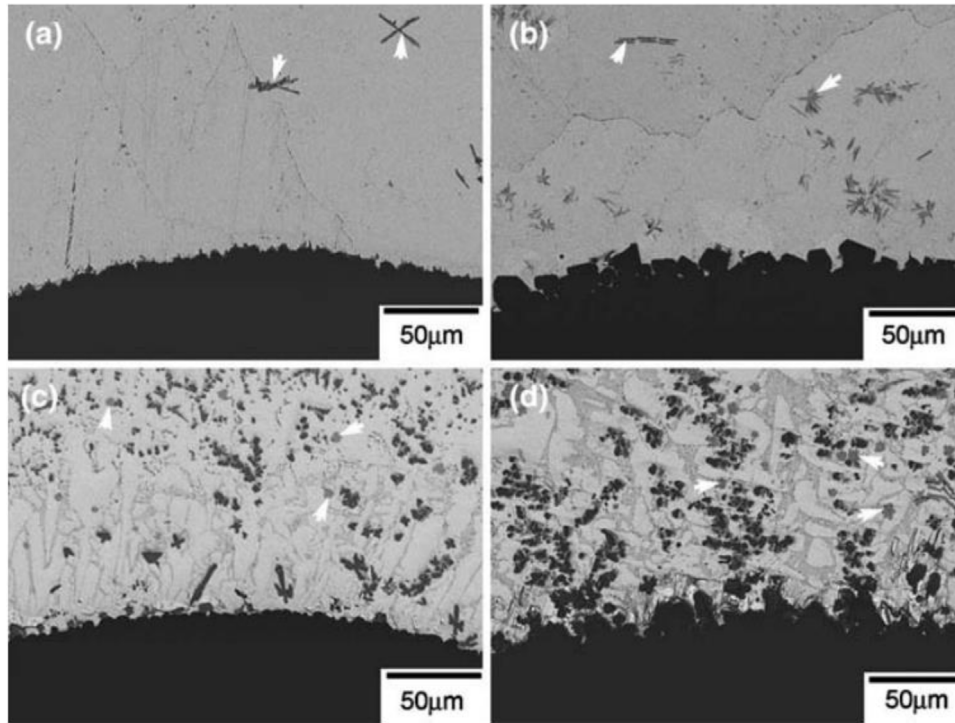


Fig. 12. Microstructure in the vicinity at the interface of Bi/Cu: (a) 350 °C for 120 min, and (b) 410 °C for 120 min; and Bi-11Ag/Cu: (c) 350 °C for 120 min, and (d) 410 °C for 120 min [80].

Bi and Ag does not form IMCs with Cu [78]. Cu-Bi-Ag eutectic phase can be formed along the Bi-Ag/Cu interface [79]. As shown in Figure 11, the solubility of Cu in a liquidus state Bi-Ag alloy was enhanced with Ag content increment and temperature. The Cu was dissolved into the liquid Bi-Ag, and Cu-rich needles with a composition of Cu-35Bi-5Ag precipitated without forming interfacial IMCs [59]. The Bi-Ag/Cu joints possessed comparable tensile strength with the Pb-5Sn solder on the same Cu substrate. The tensile strength is contributed by the grain boundary grooving. Figure 12 presents the interfacial morphologies of Cu interacting with Bi and Bi-Ag, respectively. Rather than forming typical IMC layers, grooving was observed at the solder/substrate interface. Furthermore, particles, marked by arrows, were seen within the solder matrix, in addition to the presence of Bi and Ag.

The mechanical property of the Bi-Ag/Cu joint is much better than the Bi-Ag/Ni joint due to the brittle NiBi_3 IMCs formed with Ni metallization [59]. Also, Bi-2.5Ag has poor wetting abilities on Ni metallization, which can be slightly improved by adding Ag content to Bi-11Ag [78]. As shown in Figures 13a and 13b, a NiBi_3 layer is the only phase formed at the Bi-2.5Ag/Ni interface after heating at 350 °C for 1 min [64]. With an increased reaction time of 5 min, a thin NiBi layer developed along the Ni metallization. At the same time, vertical cracks were observed to initiate and propagate within the NiBi_3 layer [64].

For Bi-Ag solder, currently no published literatures are found for investigating the interfacial reaction with Ag or Au finish. It can be concluded that the ENIG finish has

similar results with Ni metallization since the Au layer is very thin. It would also be interesting to understand the effect of immersion Ag finish on the interfacial reactions with Bi-Ag solder, even no formation of any IMC phases may be possible.

3.5 Zn-Al solders

Zn-Al based alloy can be a good choice for high-temperature die-attach solder because of its low cost and suitable melting range [81]. It also has good thermal/electrical conductivities and mechanical properties [52,82]. Zn-Al alloy has a ultimate tensile strength of 184-196 MPa, which is much higher than that of Pb-5Sn solders (28 MPa) [83]. The thermal conductivity of Zn-Al (100 W/mK) is also much better than eutectic Au-Sn (60 W/mK) and high-Pb solders (26 W/mK) [51]. The electrical conductivity of Zn-5Al (32-34% IACS) is also higher than that of Pb-5Sn (8.79% IACS) [83]. However, both Zn and Al are reactive metals, they are prone to the oxidation without a protective environment. Therefore, soldering with Zn-Al usually needs to be done under vacuum, inert gas, or even a formic acid atmosphere to remove Zn and Al oxides [57,84]. Upon soldering, the Zn and Al oxide layer will hinder the interfacial reaction with the substrate. Therefore, externally applied pressure may be necessary during soldering to break the oxide layer [59].

The addition of Cu to Zn-Al alloys can lead to a change in the microstructure by eliminating the brittle Zn-Al eutectic phase leading to the improvement in the mechanical properties of the joints [85]. Compared to Bi-2.5Ag and Au-20Sn, Zn-Al-Cu solders have superior

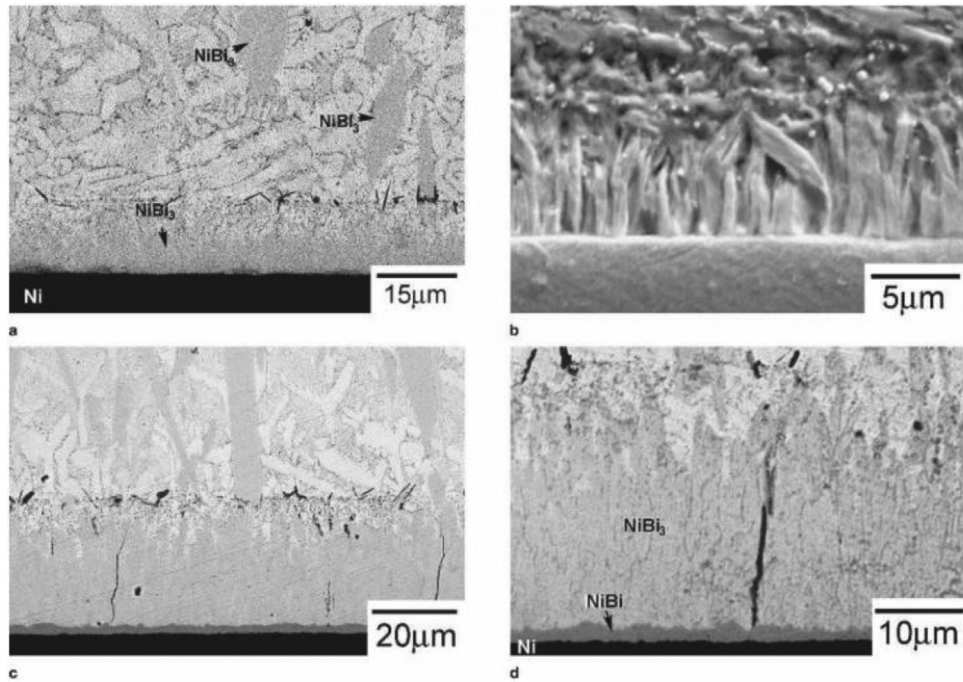


Fig. 13. Structural feature of the Bi-2.5Ag/Ni interface after reaction at 350 °C for (a) 1min.; (b) magnified structure of (a) showing that only NiBi_3 was found; for (c) 5min.; (d) magnified structure of (c) illustrating that NiBi and cracks of NiBi_3 appeared [64].

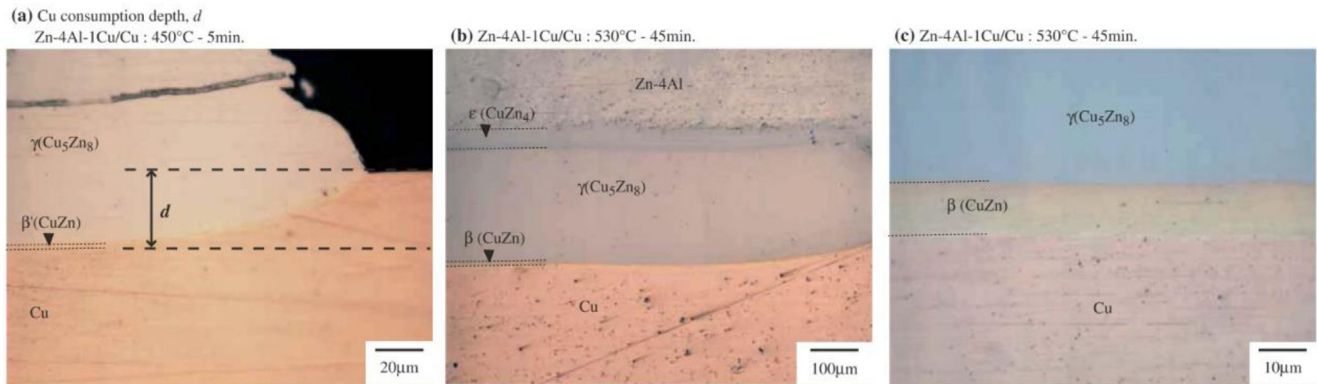


Fig. 14. Microstructures of Cu/molten Zn-4Al-1Cu (a) soldered at 450 °C for 5 min showing consumption of the Cu substrate depth d , (b) soldered at 530 °C for 45 min showing IMPs of e , c , and b , and (c) a magnified view of (b) near the interface of c , b , and Cu [53].

mechanical properties [85]. The alloy showed excellent characteristics such as high solderability, and good mechanical properties for ultrahigh temperature applications.

Zn-based solders can react actively with Cu substrate, forming excessive Cu-Zn IMCs at elevated temperatures to make the joints brittle and easy to fracture [86]. To solve this issue, a Ni deposit can be utilized between the Zn-based solder and Cu substrate to prevent formation of Cu-Zn IMCs [86]. However, the wetting ability on Ni metallization is lower than that on Cu substrate [87]. The contact angle of Zn-Al-Mg-Ga solder on Cu substrate is around 3°, which is lower than the 5° on Ni metallized Cu substrate [87].

The interfacial reactions between Cu/Zn-4Al and Cu/Zn-4Al-1Cu, including the consumption of the Cu substrate and the growth of intermetallic phases (IMPs)

during soldering and aging, were examined (Fig. 14). It was found that the consumption of the Cu substrate and the growth of IMPs during soldering were primarily driven by the diffusion of constituent elements. When 1% Cu was added to the Zn-4Al solder, a noticeable reduction in Cu substrate consumption was observed [53].

Figure 15 exhibited the typical microstructure of the soldered Zn-Al/Cu and Zn-Al/Ni interface. The Zn-Al/Cu interface usually has the $\text{CuZn}/\text{Cu}_5\text{Zn}_8/\text{CuZn}_4$ three IMCs layers, while Al_3Ni_2 is the only continuous IMCs layer that can be distinguished in the Zn-Al/Ni interface. The growth rate coefficient k of the Al_3Ni_2 IMC layer in Ni/Zn-Al is about $1.1\text{--}1.3 \times 10^{-8} \text{ m/s}^{1/2}$ at 420 °C. As a comparison, the $\text{CuZn}/\text{Cu}_5\text{Zn}_8/\text{CuZn}_4$ three layers have

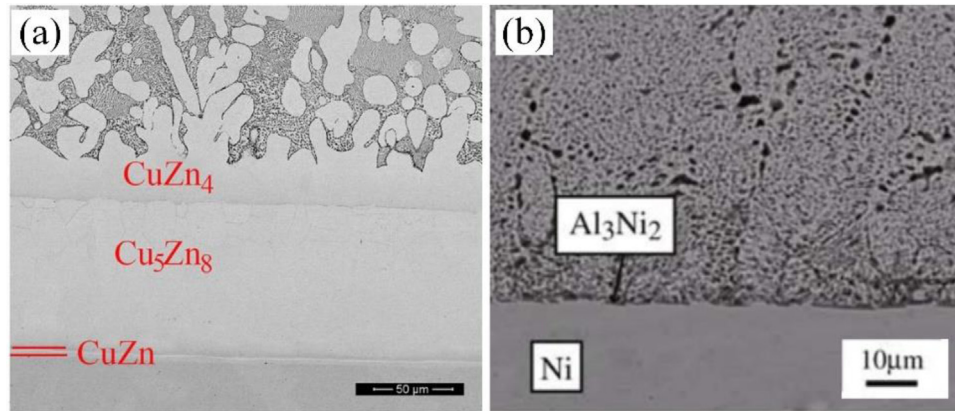


Fig. 15. Microstructures of soldered (a) Zn-3Al/Cu (500 °C, 3 min) [47] and (b) Zn-Al/Ni (420 °C, 5 min) interfaces [88].

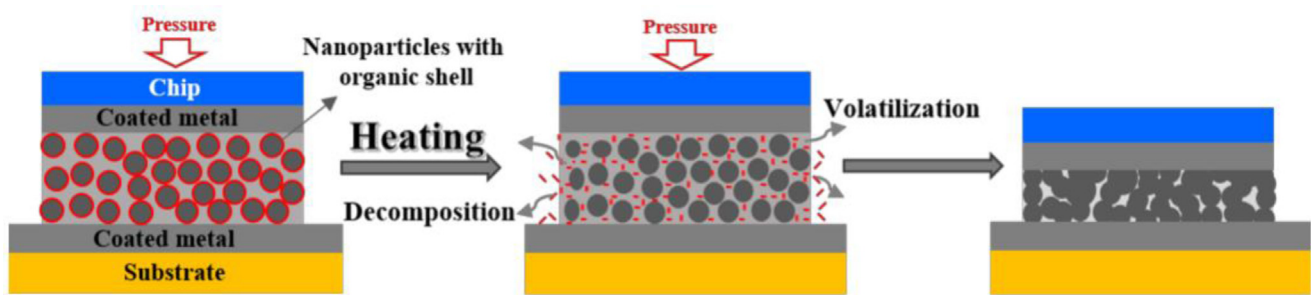


Fig. 16. Illustration of the sintering-bonding process using polyol-based Ag NPs [95].

the higher growth rate coefficients of $7.10 \times 10^{-8} + 7.10 \times 10^{-8} \text{ m/s}^{1/2}$ at 420 °C, which confirmed the diffusion barrier effect of Ni metallization.

The interfacial reactions in the soldering of Zn-Al on Ag or Au finish are currently unavailable in the literature. Tomasz et al. [89] investigated the solderability of Zn-5Al eutectic alloy with 0.5–1.5 at.% Ag addition on Cu substrate. It has been found that Ag addition inhibits the growth of Cu-Zn IMCs, since Ag_5Zn_8 has stable thermodynamic properties compared to Cu_5Zn_8 , which means Ag finish can potentially act as a diffusion barrier layer to retard the IMC formation at the interfaces. Although Ag or Au finish can protect the Cu substrate from oxidation, a protective atmosphere is still a necessity for Zn-Al soldering due to its poor oxidation and corrosion resistance. Therefore, the uses of Ag or Au finish for Zn-Al soldering may not be such beneficial.

3.6 Nano Ag and other metal pastes for sintering

Nano or micro-scale metal particles can be sintered at temperatures below their melting points due to their larger surface energy [59], which has attracted growing attention in recent years for electronic packaging applications [90]. The nano-Ag has already been applied in electronics packaging, because of its excellent properties of anti-oxidation, stability and low-temperature atomic diffusivity [91]. Low-pressure or even pressure-less nano-Ag sintering in the ambient atmosphere is available due to its small size

effect and anti-oxidation property [92]. Figure 16 illustrates the bonding process using nano Ag pastes in air. During sintering, no phase transition occurs, as the bonding is exclusively driven by solid-state diffusion [60]. A protective atmosphere may be necessary for nano-Ag sintering on Cu substrate or Ni metallization due to the oxidation, which is an extra cost during the manufacturing process. Apart from nano-Ag paste, various nano or micro particle metal pastes have been developed for electronic packaging [60,93,94]. Some properties of selected sintering solutions are listed in Table 5.

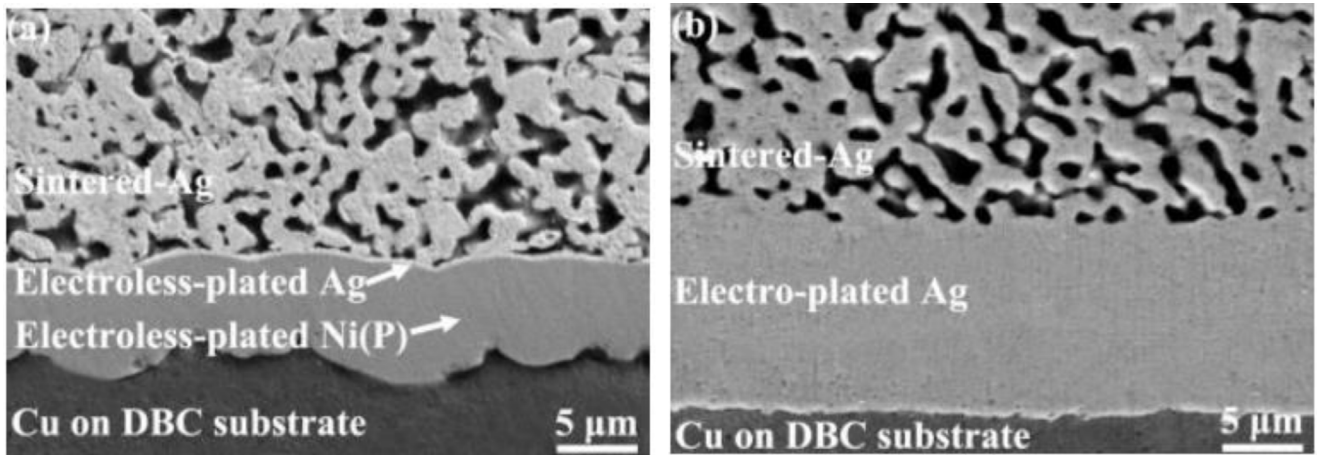
It should be noted that no IMCs are produced during nano-Ag sintering on the metallizations mentioned above. Solid solutions are formed during the interfacial reactions, which highly depends on the interdiffusion rate between nano-Ag and metallization [24]. The atomic inter-diffusivity among Ag, Au, Cu and Ni is shown in Table 6. It has been concluded that the interfacial adhesion of nano-Ag sintering highly relies upon the types of metallization [24]. The chemistry and microstructure of metallization can influence the atomic interdiffusion rates during the sintering process. The diffusivity of Ag in Ag is over 100 times higher than in Ni. As a result, Ag atoms diffuse more quickly through electro-plated Ag than through electroless-plated Ni(P)/Ag, leading to stronger interfacial adhesion on the electro-plated Ag (Fig. 17). This difference in diffusivity helps explain why electro-plated Ag demonstrates a die-shear strength roughly 10 MPa greater than that of electroless-plated Ni(P)/Ag [96].

Table 5. Common serving and operating temperatures of selected sintering metal pastes [60,93].

Materials	Serving temperature (°C)	Processing temperature (°C)
Nano Ag	961	250
Ag-Sn	>600	250
Ag-In	>880	175
Nano Cu	1064	300
Cu-Sn	>415	280
Cu-In	>307	180
Sn-Ni	>400	300

Table 6. Atomic interdiffusion behaviour among Cu, Ni, Ag and Au [24].

Diffusion direction	Diffusivity (300 °C, cm ² /s)	Diffusion length at 300 °C for 10 min (nm)
Ag into Ag	5.99×10^{-18}	0.60
Ag into Au	3.42×10^{-17}	1.43
Au into Ag	4.67×10^{-19}	0.16
Ag into Ni	2.43×10^{-20}	0.04
Ni into Ag	2.67×10^{-20}	0.04
Cu into Ag	3.09×10^{-18}	0.43
Ag into Cu	1.14×10^{-18}	0.29

**Fig. 17.** SEM microstructures of sintered Ag bond-line at 240 °C on: (a) electroless-plated Ni(P)/Ag and (b) electro-plated Ag metallization [96].

Nano-Ag sintering on Ag finish has been regarded as a preferable route, which can provide reliable bonding above 40 MPa [24]. On Au metallization, Kirkendall voids can be formed at the Ag-Au interface due to the 100-fold interdiffusion rate difference between Au and Ag, which results in a lower shear strength compared to that on Ag finish [97]. The Ag-Ni diffusivity is 100-fold lower than the Ag-Ag interface, which results in 10 MPa

lower shear strength [24]. For the ENIG substrates, the shear strength of these joints was significantly lower compared to that of Ni/Au joints. A continuous Ag/Au layer formed along the interface, and a delamination zone developed adjacent to the Ag/Au layer (Fig. 18).

Large amount Ag atoms of the nano Ag particles adjacent to the ENIG, which are going to be diffused to the Au finishes during the sintering process [76]. Figure 19

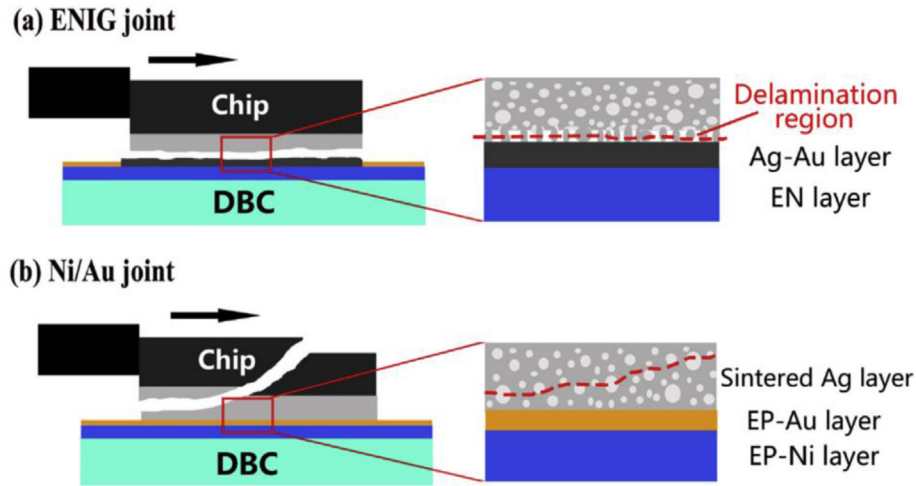


Fig. 18. Schematic illustrations of fracture modes for (a) ENIG and (b) Ni/Au joints [36].

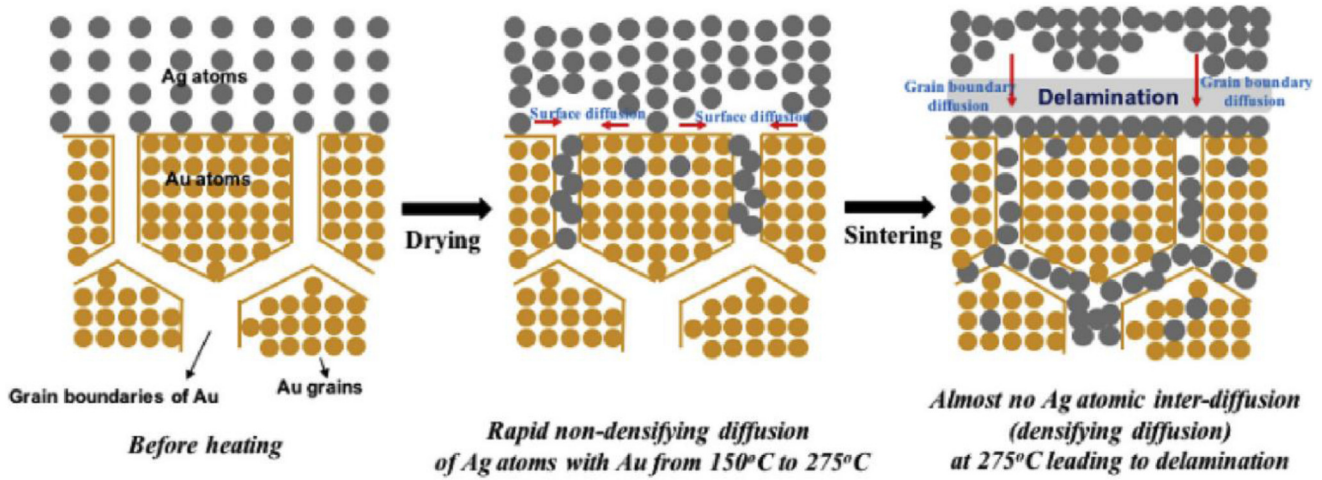


Fig. 19. Schematic diagram of atomic inter-diffusion among sintered Ag and ENIG [76].

Table 7. Interfacial reaction products between metallization and high-temperature interconnection materials.

Surface finish	Pb-5Sn	Bi-2.5Ag	Au-20Sn	Nano Ag	Zn-5Al
Bare Cu	Cu ₃ Sn [62]	Cu-35Bi-5Ag solid solution [64]	AuCu/(Au, Cu) ₅ Sn [75]	Cu-Ag solid solution [20]	CuZn/Cu ₅ Zn ₈ /CuZn ₄ [53]
Ni finish	Ni ₃ Sn ₄ [63]	NiBi/NiBi ₃ [64]	(Ni, Au) ₃ Sn ₂ [74]	Ni-Ag solid solution [20]	Al ₃ Ni ₂ [88]
Ag finish	Ag ₃ Sn [64]	Not reported	Not reported	Ag [20]	Not reported
ENIG	AuSn ₄ [34]	Not reported	(Ni, Au) ₃ Sn ₂ [67]	Au-Ag solid solution [20]	Not reported

illustrates that when an excessive amount of Ag atoms diffuses rapidly into the Au finish, the remaining Ag atoms from the Ag-NPs near the interface are insufficient to densify into a strong sintered Ag joint. This is because the atomic diffusivity of Ag to Ag is much lower than that of Ag to Au at temperatures above 150°C.

A similar phenomenon can be observed in Cu sintering. The shear strengths of nano Cu paste on Cu and Ni metallization can be in the range between 54 MPa and 60 MPa, which is higher than that on Ag (36 MPa) and Au finish (25 MPa) [93]. After thermal aging, the shear strength of the joints formed on Ni and Cu finish increases,

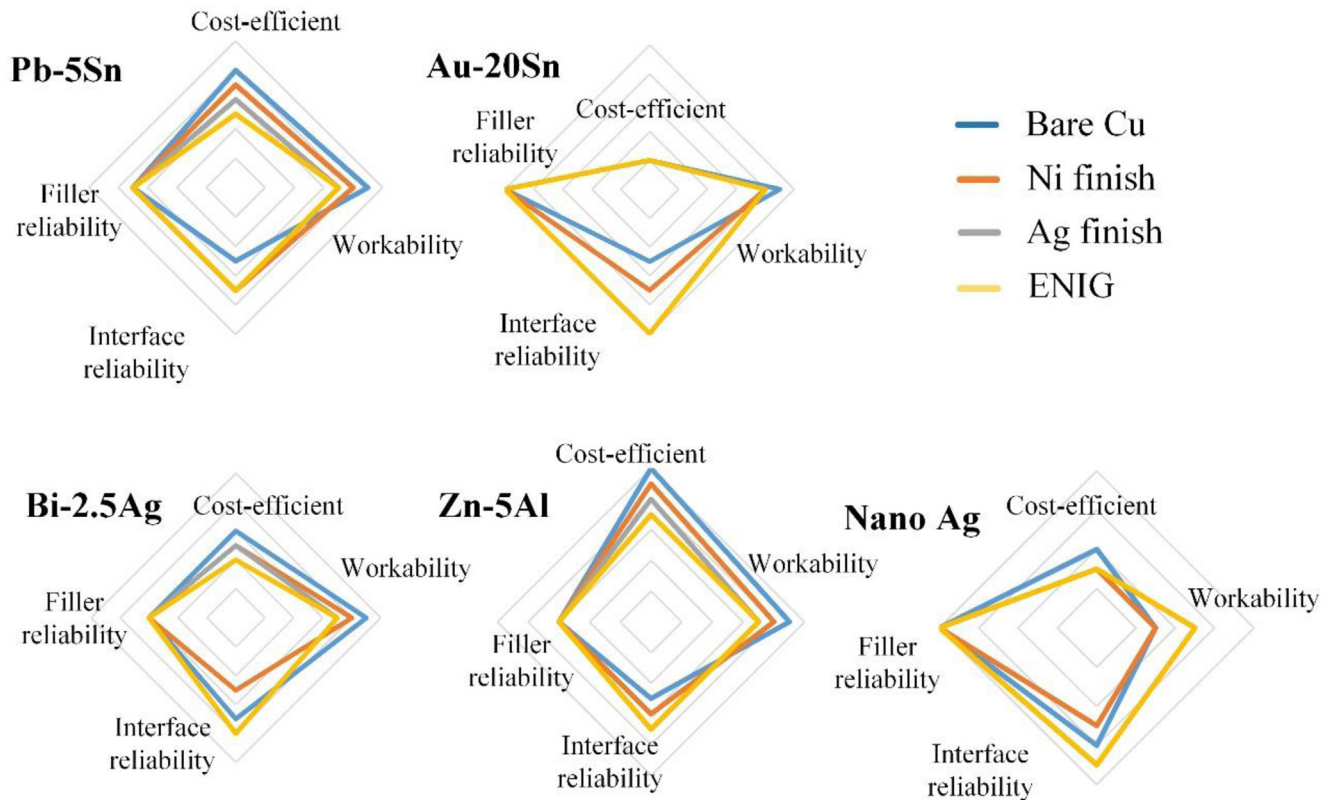


Fig. 20. Radar map of Pb-5Sn, Au-20Sn, Bi-2.5Ag, Zn-5Al and nano Ag.

but it decreases on Ag and Au metallization. This is because of the interdiffusion coefficient difference between Cu paste and the Ag or Au finish, which results in the porous interface in the sintered Cu/Ag and Cu/Au joints. In contrast, the interfacial microstructures between the sintered Cu and the Ni or Cu layer remain stable after aging since the interdiffusion is relatively minor [93]. It can be concluded that the equivalent atomic diffusion rates between sintering paste and surface finish are beneficial for the enhancement of bonding strength of the joints. In particular, the nano-Ag sintering on Ag finish and nano Cu sintering on Cu substrate tend to form the most stable joint microstructure owing to the similar mutual atomic diffusion rates.

4 Conclusion

This review article offers a comprehensive overview of the interfacial reactions between high-temperature interconnection filler materials and surface finishes in die-attach applications. It emphasizes the critical role of metallization on both the die and substrate, which is essential for joint formation and ultimately determines the reliability of die-attach under high-temperature operating conditions.

Based on the discussion above, Table 7 summarizes the resultant products of interfacial reactions between surface metallization and five commonly used high-temperature die-attach materials. To evaluate the effectiveness of different metallizations, radar maps for these five

die-attach materials are presented in Figure 20. Each combination is assessed across four indices: processability, cost-effectiveness, interface reliability, and filler reliability. Processability reflects the feasibility and applicability of the bonding processes; this index will be rated higher if additional metallization or protective atmospheres are required. Cost-effectiveness is lower when precious metals, such as Ag and Au, are involved. Filler reliability is based on the properties of the filler materials, while interface reliability is determined by the characteristics of the interfacial reaction products.

The effects of these metallizations on interfacial reactions exhibit distinct characteristics when novel interconnection materials are utilized. The main conclusions and recommendations are summarized as follows:

- In contrast to a bare Cu substrate, Ni metallization acts as an efficient diffusion barrier, especially for high-Pb, Zn-Al, and Au-based solders. Without nickel, Cu can rapidly dissolve into molten solders during the soldering process, which raises concerns about the reliability of the resulting joints and interfaces.
- The advantages of using silver and gold finishes for high-Pb and Zn-Al solders are not particularly compelling due to the additional costs involved. High-Pb solders already exhibit good wetting properties; however, a protective atmosphere is necessary during soldering with Zn-Al solders, even when using silver or gold finishes. For the costly gold-based solders, an ENIG coating is a viable option to facilitate fluxless soldering, thanks to the non-oxidizing and highly protective nature of gold in air.

- Ni metallization is not a favorable option for soldering with Bi-Ag solders. Bi-Ag does not form IMCs with Cu substrates, but can form brittle NiBi_3 with nickel metallization, significantly compromising the mechanical integrity of the joints. In contrast, Bi-Ag can react with silver to create a stable solid solution on silver finishes without the formation of IMCs between bismuth and silver. Further research is needed to explore the solderability of Bi-Ag on silver surface finishes.
- Solid solutions are the primary products formed during nano-silver and Cu sintering, rather than IMCs. To achieve higher shear strength and stabilize the joint microstructure, similar atomic interdiffusion coefficients between the metal paste and metallization are desirable. For example, nano-silver sintering on a silver finish represents the most logical and practical process route. However, a protective atmosphere is likely necessary for Cu sintering on a Cu substrate, given its strong tendency to oxidize.

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Author contribution statement

Conceptualization, Canyu Liu; Methodology, Canyu Liu; Writing – Original Draft Preparation, Canyu Liu; Review & Editing, Changqing Liu; Supervision, Changqing Liu; Project Administration, Changqing Liu.

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