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Review Article

Bi-Ag as an Alternative High Temperature Solder

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ABSTRACT

The search for a high temperature lead-free solder replacement for high temperature leaded solder eutectic alloy has been an evolving process as the threat of a regional lead ban became a reality in July 2006. The advantages and disadvantages of lead-free solder in terms of manufacturing, performance and reliability have been increasingly revealed through companies' Research and Development (R&D), industrial consortia and university researchers. Materials and component design are the primary criteria to focus on the development for the current generation of high temperature lead-free solder alloys. According to the current status of high temperature lead free soldering, there are many unsolved technical problems such as explanation on the lift-off phenomenon, establishment of high temperature lead-free plating technology, construction of a database of physical properties (solder, parts, PCBs), standardization of high temperature solder materials evaluation technology, and most importantly, the best candidate material for high temperature solder. Clearly, high temperature soldering is one of the unsolved problems of the century in lead-free soldering. Moreover, most of the questions still remain unanswered by researchers. This paper reviews research conducted on the Bi-Ag solder alloy, which is one of the candidate alloys that has been proposed as an alternative for high temperature lead-free solder.

Keywords: Lead-free Solder, Bi-Ag Solder Alloy, Intermetallic Compound (IMC), Microstructure

INTRODUCTION

Soldering is a metallurgical process for joining metal parts and it uses molten filler metal to wet the surfaces of a joint that provides a conductive path required in achieving a connection from one circuit element to another. It is also important for interconnection in both level 1 (IC Packaging)

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and level 2 (mounting of electronic components onto printed circuit boards) processes of the modern electronics industry. Hence, in order to produce a high quality high temperature soldering process, it is important to understand the fundamental of solder and soldering. The soldering process can be divided into three important factors that must be considered, namely, spreading, base metal dissolution, and formation of intermetallic compound layer (Lea, 1988).

Lead-free solder is defined as elimination of lead in solder alloys system because of the response to the concerns for the environment and human health. Environmental Protection Agency (EPA) already mentioned lead as one of the top 17 chemicals that poses the greatest threats to human life and the environment (Wood *et al*., 1994). Disposed lead can leach into drinking water, posing a severe health risks to human. Among the common types of lead poisoning are alimentary, neuromotor and encephalic (Ir. Sax, 1984). Lead poisoning can be detected when its level in blood exceeds 50 mg/dl of blood (Napp, 1995). Occupational Safety and Health Administration (OSHA) requires that workers have no more than 50 mg/dl of lead in their blood and recommends their workers to maintain blood level below 30 mg/dl if they plan to have children (EPA, 1991). Wave soldering produces dross on the molten solder and about 90% of dross can be refined to pure metal for reuse, but the remaining acts as waste product (Nriagu *et al*., 1988).

Attempts to recycle lead in printed wiring boards of consumer electronic products have not been successful. Therefore, Japan has required that all new electronic products be lead-free solder from January 2005, while the European Union introduced legislation to ban lead from electronic products by 1st July 2006. Nevertheless, the United States government has not yet legislated against the use of lead in electronic products. In response to the new legislation, most major electronic manufacturers, including those in the U.S., have stepped up their search for the alternatives to leaded solders. Even when the use of lead in electronics industry appears to be minimal, the potential for lead exposure cannot be ignored (Monsalve, 1984).

Although Sn-Pb can be replaced by lead-free solder for low temperature applications, there is no lead-free solder available in the market that can replace the high temperature leaded solders. Most of the internal joining such as in the first level interconnection flip chip uses 90 to 95 wt. % of lead (Katsuaki *et al*., 2009). Stated in the RoHS (The Restriction on the Use of Hazardous Substances) regulation, the law gave exemptions to the industry by allowing the use of solder with above 85% lead. The challenge faced towards substituting leaded solder is finding the right combination of alloy that can withstand the high temperature working condition with the reflow peak temperature and must not overshoot the temperature range that may damage the substrate (warpage).

High-temperature solders have been widely used in various applications. It is not only used as a die-attach solder, but also in the aircraft industry, space satellite, automotive, oil and gas well explorations, assembling optical components, automobile circuit boards, circuit modules as step soldering and many more. Most importantly, it can produce reliable interconnections, in which the high-density packaging technology and high-service temperature are needed. Important characteristics of the high-temperature solders are good wetability, high ductility, low shear modulus and good resistance to thermo-mechanical fatigue. Due to environmental reasons, the development of lead-free solders has become an important issue and until now, only a few alloy candidates have been proposed for the lead-free high-temperature solders such as

Au-Sn, Bi-Ag, and Zn-Al based alloys. The Bi-Ag alloy is the most suitable because it exhibits a high melting temperature, similar hardness with Pb-5Sn and lower cost as compared to Au-Sn (Yaowu Shi *et al*., 2009). Meanwhile, silver element is widely used in hybrid microelectronics because the cost is lower compared to gold and palladium. Moreover, silver can be processed in air without having to concern about oxidation that affects its electrical resistivity as Ag_2O is conductive. This unique property gives high electrical and thermal conductivity and exhibits limited fatigue.

The Bi-Ag Binary Phase Diagram

Phase diagram gives valuable information on the various phases of a substance and the conditions under which each phase exists. It is a powerful tool for developing new solder since a wide range of potential alloys can be easily evaluated in theory (Ursula, 2002). Bi-Ag phase diagram in Fig.1 shows that the eutectic point is 262.5°C when the composition is at 97.5 wt. % Bi and 2.5 wt. %Ag.

As in the soldering process, eutectic solder material is normally used because of its lower temperature that reduces the likelihood of component damage, as well as lower dross formation during the soldering process due to the one point melting phenomena. The Bi-Ag alloy system exhibits a higher melting point at other wt. % combination. A further investigation by Lalena has shown that increasing the Ag content of the Bi-Ag alloy to 11 wt. %, promotes an increase in thermal conductivity and ductility (Lalena *et al*., 1980).

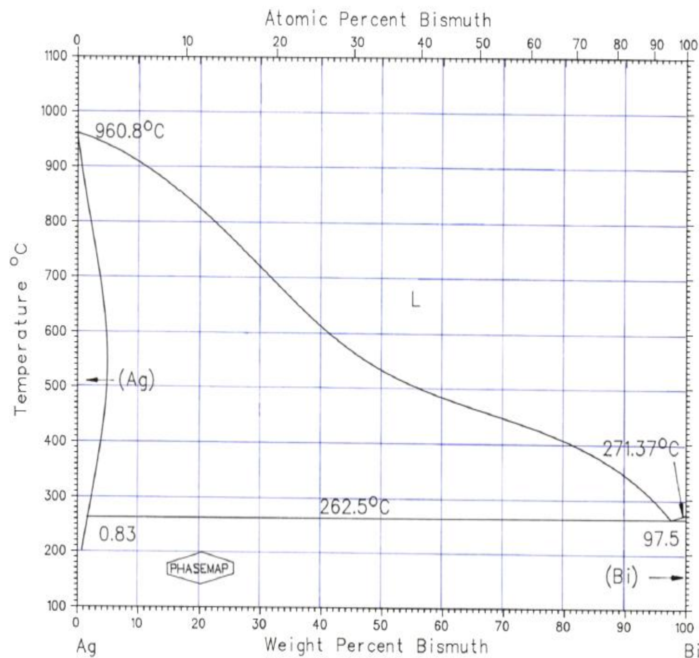

Figure 1: The Bi-Ag Binary Phase Diagram (Elliott *et al*., 1980)

Melting Temperature Modifications

The solidus line of the Bi-Ag alloy solder remains almost constant at a temperature of 262.5°C according to the phase diagram. The temperature between the liquidus and solidus line increases linearly with Ag wt. %. Yaowu Shi, in Table 1, shows that addition of small amounts of rare earth Ce will slightly decrease the melting temperature. This is important because, for high lead solder replacement, the lead-free solders must have solidus temperature higher than 260°C so that it will not melt during further assembly process or during usage (Katsuaki *et al*., 2009). The Bi-Ag system may have fulfil the requirement of high temperature solder even when the solidus temperature of the Bi-Ag alloys is lower than that of Pb5Sn. However, to improve the properties of high temperature lead free solders, the rare earth (RE) elements are used. Other than reducing the melting temperature, it is also known as a surface-active element that promotes refinement of microstructure, alloying, and metamorphosis of inclusions. The addition of the rare elements on the lead free solders alloys has been widely studied by researchers (Xia *et al*., 2002; Chen *et al.*, 2002; Wu *et al.*, 2007). The melting temperatures of Bi-Ag, Bi-Ag-RE, and Pb-Sn solder are as shown in Table 1:

TABLE 1
Melting temperature of Bi-Ag, Bi-Ag-RE and Pb-Sn solder (Yaowu Shi *et al*., 2009)

Solder	Liquidus (°C)	Solidus (°C)
Bi2.5Ag	262.6	260.9
Bi2.5AgRE	262.3	260.9
Bi5AgRE	301.5	260.9
Bi7.5AgRE	334.5	260.7
Bi10Ag	380.6	261.4
Bi10AgRE	378.2	260.8
Pb5Sn	311.6	310.6

Intermetallic Compund (IMC)

The diffusion layer between solder and substrate is known as intermetallic compound (IMC). In the soldering theory, the most important part that must be considered during a soldering process is intermetallic formation. Soldering does not only involve physical dissolution, but it is also a chemical reaction process when the solder wets on the substrate (Humpston *et al*., 1993). In other words, without intermetallic compound formation, soldering can not take place. Intermetallic compounds will enhance solder wetting on the substrate and reduce the dissolution rate of substrate in solder through the diffusion barrier role of the intermetallics layer. Intermetallic formation strictly follows stoichiometric ratios of elements, where some elements have high affinity for each other to even deny bonding with other elements (Lea, 1988).

Microstructure of Bi-Ag/Cu Interface and Tensile Behavior of Cu/Bi-Ag/Cu Joints

Fig.2 shows the rapidly solidified structure of Bi-Ag pellets examined using Scanning Electron Microscope (SEM). Non-equilibrium solidification structural features were identified. The Bi-

2.5Ag sample microstructure contains coarse proeutectic Bi and fine Bi-Ag eutectics, while the hypereutectic microstructure comprises of dendritic primary Ag, proeutectic Bi, and Bi-Ag eutectics that can be seen in the Bi-11Ag sample. In a study conducted by Jenn-Ming Song, some primary Ag were also found in the Bi-2.5Ag sample at the eutectic cell boundaries. Higher Ag wt% resulted in an increased population of the primary Ag and proeutectic Bi in the Bi-11Ag sample compared to the Bi-2.5Ag sample (Jenn *et al.*, 2007b).

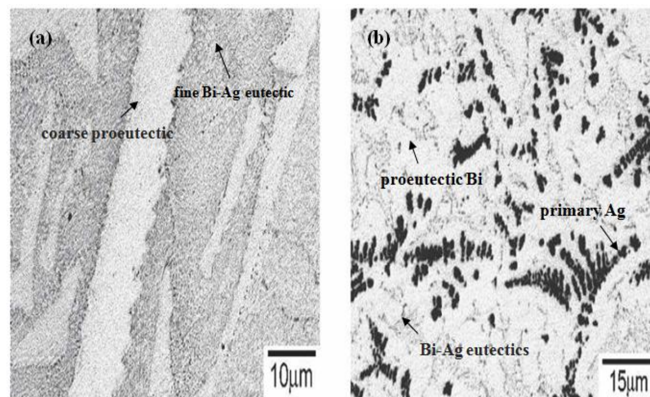

Fig.2: Microstructure of Bi-Ag solders balls; (a) Bi-2.5Ag, and (b) Bi-11Ag (Jenn *et al*., 2006)

Fig.3 shows the microstructure of the Bi-2.5Ag sample on Cu substrate after soldering for 1 min and 5 min. From the observation, there was no intermetallic compound formed but the Cu substrate became rougher, as illustrated in Fig.3a. In Fig.3a and Fig.3b, the small amount of Ag-rich compound precipitates on the Cu substrate, regardless of the reaction time. A Cu-rich phase, with acicular morphology, and the elements composition of 60 at.%Cu-35at.% Bi-5at.%Ag, were seen when the soldering time reached 5 min. Fig.2c shows the fan-like appearance of the Cu-rich needles.

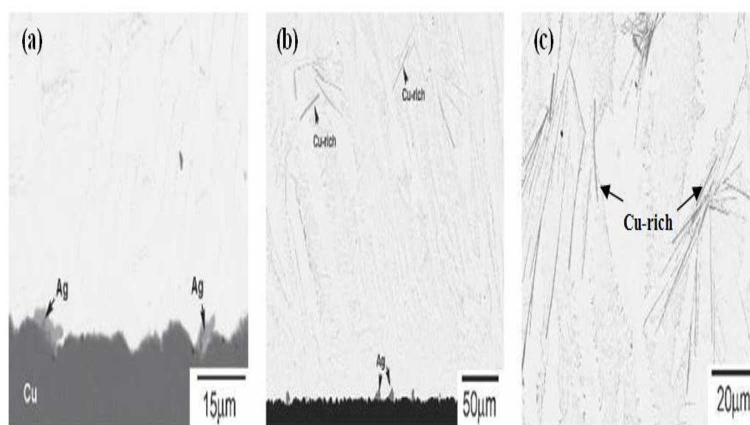

Fig.3: The structural features of the Bi-2.5Ag/Cu interface after soldering at 350°C for (a) 1 min. and (b) 5 min.; (c) Cu-rich needle within the solder after 5min (Jenn *et al*., 2006)

Fig.4 shows the structural feature of the interface between the Bi-11Ag sample and Cu substrate after the reaction at 350 °C for 1 min and 5 min. There was a similarity found between the structure and Bi-2.5Ag interface, except for the appearance of massive primary Ag and Cu-Bi-Ag needles already existing in the Bi-11Ag/Cu sample even after soldering at 350°C for only 1 min, as illustrated in Fig.4a.

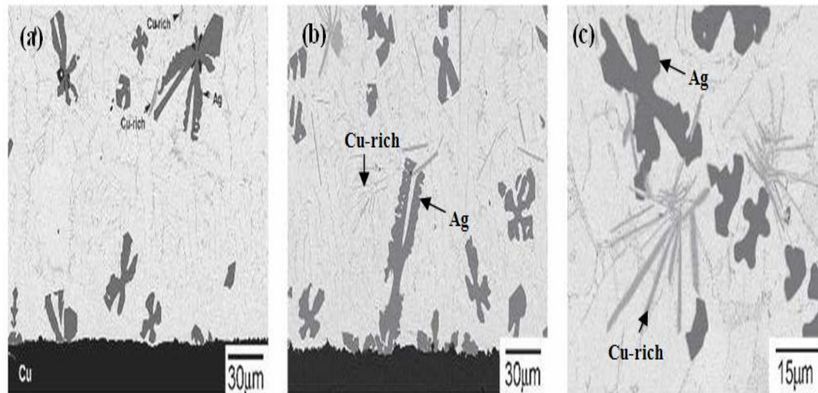

Fig.4: The structural feature of the Bi-11Ag/Cu interface after the reaction at 350°C for (a) 1 min (b) 5 min.; (c) primary Ag and Cu-rich needle within the solder (Jenn *et al*., 2006)

Fig.5 shows the magnified images of the grooved grain boundaries at Bi-11Ag/Cu interfaces, indicating the dissolution of solder at the Cu grain boundaries. The molten solder tended to penetrate into the Cu grain boundaries with the prolonged reaction time. The Ag addition accelerated the grain boundary grooving of Cu by molten solder (Jenn *et al*., 2007a).

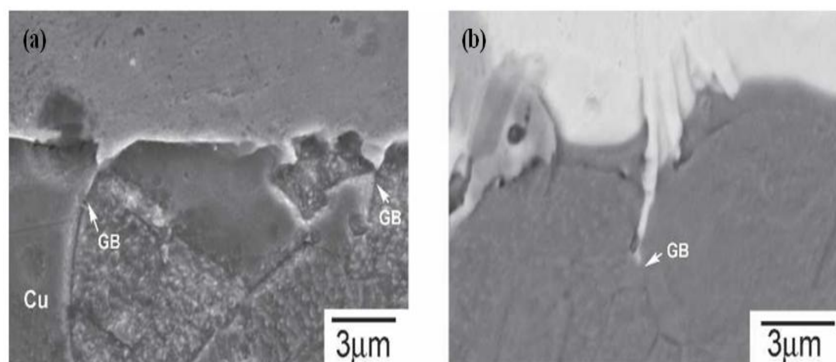

Fig.5: The grooved grain boundaries at the Bi-11Ag/Cu interface for varying reaction times: (a) 1 min. and, (b) 5 min. GB: grain boundary (Jenn *et al*., 2006)

Yaowu Shi, in his study, confirmed the findings from Jenn in 2006. He conducted a study using Bi-2.5Ag and Bi-10Ag on Cu substrate for 30 min. The microstructure of Bi-2.5Ag and Bi-10Ag solder joints shown in Fig.5 show that the Bi-2.5Ag sample mainly consists of fine Bi-Ag eutectics. Meanwhile, the non-equilibrium solidification of soldering process causes the

primary Ag particles to be found only at some visual fields and often located at the eutectic cell boundaries. However, Bi-10Ag alloy consists of a hypereutectic microstructure comprising dendritic primary Ag and Bi-Ag eutectics. Yaowu Shi also confirmed the formation of Cu-rich acicular morphology in his work. Once again, no intermetallic compound (IMC) formed at the interface between the Bi-Ag solder and substrate.

Fig.6b and Fig.6c clearly show many Ag-rich phases that are formed near the solder side at the interface. The formation of Cu-rich acicular phase results in the Ag-rich phase formation near the interface of Bi-Ag solder.

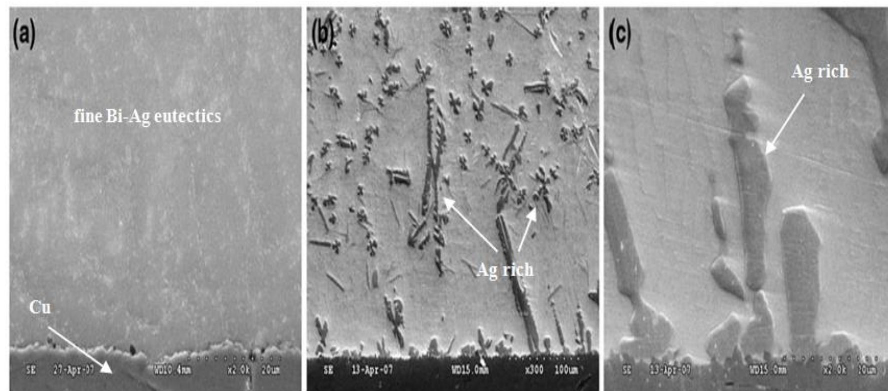

Fig.6: The microstructure of Bi-Ag solders joints; a) Bi-2.5Ag, b) Bi-10Ag, (c) Bi-10Ag with high magnification (Yaowu Shi *et al*., 2009).

The fracture surface of the Bi-11Ag/Cu sample in a study conducted by Jenn showed that fracturing mostly occurred within the solder, as shown in Fig.7. However, the Bi-2.5Ag/Cu sample, with a relatively low joint strength, showed a large area of exposed Cu, indicating an interfacial fracturing (Jenn *et al*., 2006).

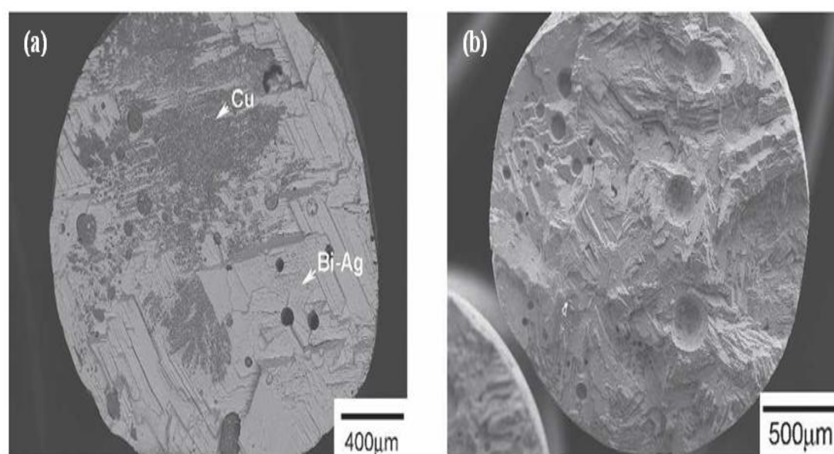

Fig.7: The tensile fracture surface of the Cu/Bi-Ag/Cu joints; (a) Bi-2.5Ag (backscattered electron image), and (b) Bi-11Ag (Jenn *et al*., 2006)

Fig.8 focuses on the cross-sectional structure and a magnified image of the fractography. Fig.8c and Fig.8d indicate that the grain boundary grooving of Cu was more pronounced in the case of the Bi-11Ag/Cu interface. It is also shown that the degree of grain boundary grooving at the Bi-11Ag interface is higher compared to the Bi-2.5Ag interface because the addition of Ag content in molten solder enhances the grooving behaviour at grain boundaries (Jenn *et al*., 2006). The surface of exposed Cu substrate is smoother for the Bi-2.5Ag interface, as illustrated in Fig.8a and Fig8.b.

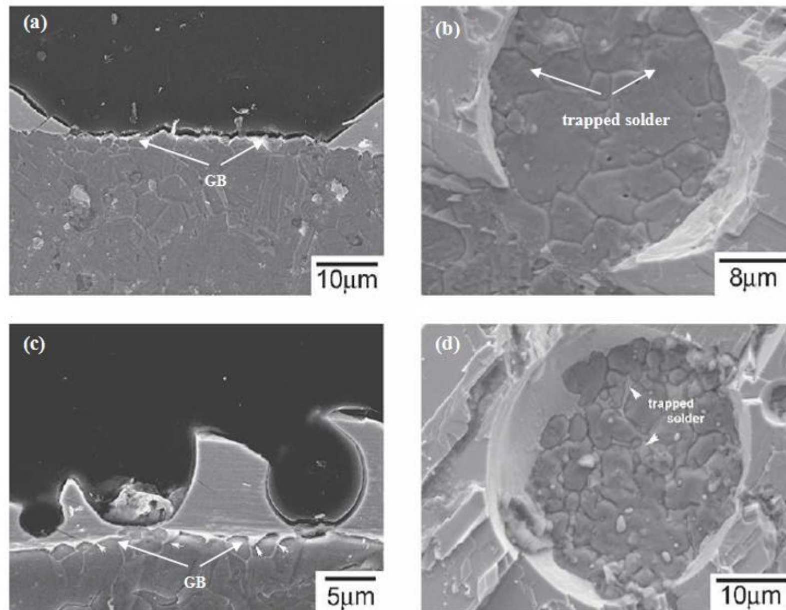

Fig.8: The cross-sectional image showing grooved grain boundaries on the exposed Cu substrate of fractured Cu/Bi-Ag/Cu joints: (a and b) Bi-2.5Ag, (c and d) Bi-11Ag. Arrows in (c) indicate grooved grain boundaries (Jenn *et al*., 2006)

Microstructure of Bi-Ag Solder Joints with Addition of Rare Earth (RE) Element

The effects of different Ag contents and the addition of rare earth element on the microstructure of Bi-Ag solder joints are displayed in Fig.9. In his work, Yaowu Shi describes that a higher Ag content gives an increase in the formation of dendritic primary Ag and acicular Cu-rich phase in the solder but the microstructure of the Bi-Ag solders has no obvious change with the addition of 0.1wt % rare earth element such as Ce in the solder. An acicular Cu-rich phase and the coarse Ag-rich phase are obviously refined, and this indicates that the needle-like structure is shortened by the addition of rare earth element. A few other research studies have confirmed that the addition of RE element can help to refine the microstructures of the solders, as seen in Sn-based solders such as Sn-Ag-Cu (Chen *et al*., 2002), Sn-Ag-Bi (Xia *et al*., 2002), and Sn-Bi (Dong *et al*., 2008). The unique properties of the rare earth element play an important role during the solidification process that will affect the microstructure of alloys (Wu *et al*., 2007; Zhang, 2006). The RE element tends to agglomerate at the interfaces of the primary phases

and at the grain boundaries during solidification. This phenomenon will reduce the interfacial energy and also refine grain size because RE acts as a surface active element.

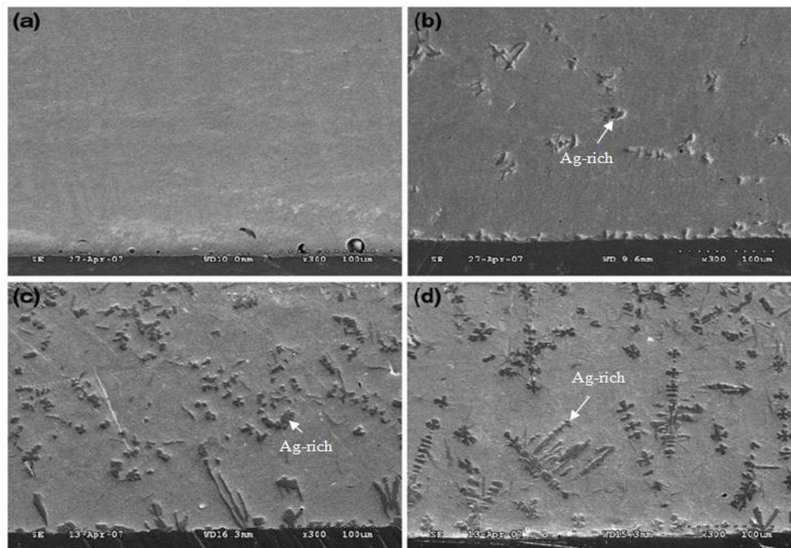

Fig.9: The effect of rare earth addition on microstructure of Bi-Ag solders joints; (a) Bi-2.5Ag-RE, (b) Bi-5Ag-RE, (c) Bi-7.5Ag-RE, (d) Bi-10Ag-RE (Yaowu Shi *et al*., 2009).

Microstructural Evolution during Long-Time Soaking

Fig.10a and Fig.10b display the magnified microstructure of the grain boundaries grooves formation at the interface of pure Bi/Cu and Bi-11Ag/Cu after a short period of soaking time at 350°C for 10 min and 410°C for 120 min, respectively. It is shown that a greater degree of grain boundaries grooving appeared when a higher temperature and an extended reaction time were used, as illustrated in Fig.10c and Fig.10d (Jenn *et al*., 2007a). The increasing depth of the grain boundary grooves is also dependent upon the addition of Ag related with the dissolution of the grain boundary, which is important in the interfacial reaction between Bi-Ag solders alloy and Cu substrate.

In a similar study by Jenn in 2007, soaking Cu wires in molten Bi and Bi-Ag alloys led to newborn phases in the solder with variable soaking times and temperatures (see Fig.11). The newborn phases compound elements are identified in Fig.12.

Fig.12 displays the newborn phase identified from the reaction of dissolved Cu and molten solders at 380°C for 120 min. In the interaction between pure Bi and Cu, acicular Cu-Bi phase with the composition of 59.6at. %Cu-40.4at. %Bi gathered with a fanlike appearance, as illustrated in Fig.12a and Fig.12b. Meanwhile in the Bi-11Ag and Cu wire samples, the Cu-Bi phase exhibited a round appearance and contained a certain amount of Ag (the composition was 56.3 at%Cu-39.2at. %Bi-4.5at.%Ag), as shown in Fig.12c and Fig.12d. A comparison between Fig.12a and Fig.12c reveals that the amount of Cu-Bi phase in the Bi-11Ag solder is larger than that of pure Bi because the Ag element will enhance the formation of Cu-Bi phase that reduces the Cu concentration in liquid Bi. Besides, the Cu-Bi phase has a round appearance in

Bi-11Ag solder alloys because the small amount of Cu has been replaced by the Ag element that modifies the fanlike appearance to round appearance.

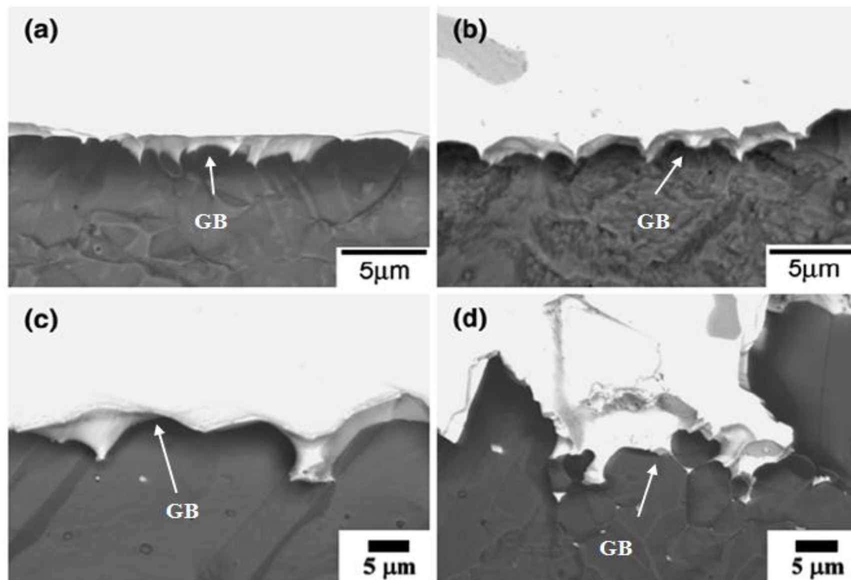

Fig.10: The interfacial morphologies of the samples after reaction under different conditions; (a) pure Bi/Cu at 350°C for 10 min, (b) Bi-11Ag/Cu at 350°C for 10 min, (c) pure Bi/Cu at 410°C for 120 min, and (d) Bi-11Ag/Cu at 410°C for 120 min (Jenn *et al*., 2007a).

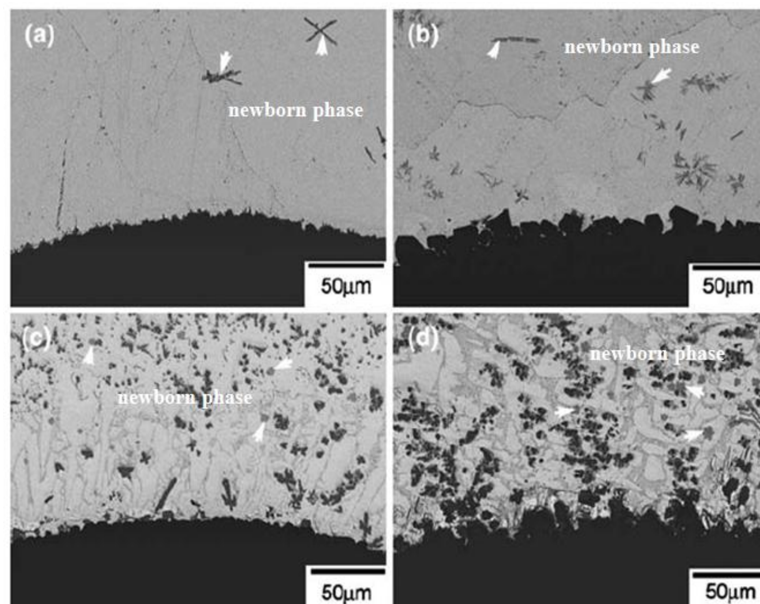

Fig.10: The microstructure in the vicinity at the interface of Bi/Cu; (a) 350°C for 120 min, and (b) 410°C for 120 min; and Bi-11Ag/Cu: (c) 350°C for 120 min, and (d) 410°C for 120 min (the gray newborn phase is indicated by the arrows) (Jenn *et al*., 2007a).

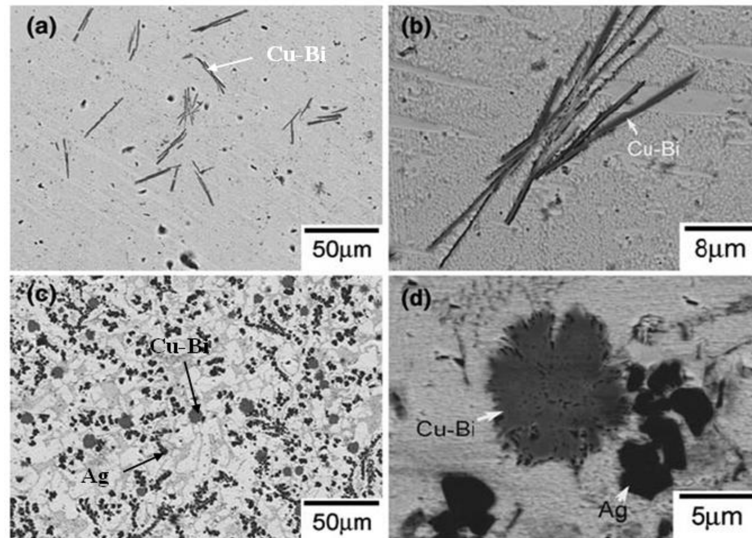

Fig.11: The micrographs of pure Bi and Bi-11Ag samples after the reaction with Cu at 380°C for 120 min; (a and b) pure Bi and (c and d) Bi-11Ag (Jenn *et al*., 2007a).

CONCLUSION

This paper reviews the microstructure of the Bi-Ag solder alloys at the interface and tensile behaviour of Cu/Bi-Ag/Cu joints, microstructure of Bi-Ag solder joints, with the addition of Rare Earth (RE) element and microstructural evolution during the long-time soaking. The Bi-Ag alloys showed a non-equilibrium solidification structural feature. Mostly, primary Ag, Bi grains, and Bi-Ag eutectics coexisted in the microstructure that is effective for microelectronic soldering application because primary Ag enhanced the ductility of the Bi-Ag solder at high deformation rates to avoid from crack growth (Jenn *et al*., 2007b). Unfortunately, Bi-Ag alloy has some disadvantages which include low electrical conductivity and thermal conductivity with low Ag wt%. However, electrical conductivity can be increased by increasing the Ag content more than 11 wt% (Lalena *et al.,* 2002; Jenn *et al.,* 2006; Fang *et al.,* 2008). Similarly, the Bismuth solder also possesses poor wetting on substrate such as copper (Kim *et al*., 2002) and to improve the wetting properties, the solder must be alloyed with other elements such as tin and rare earth element (Katsuaki *et al*., 2009). Meanwhile, increasing Ag wt. % will improve the ductility of brittle Bi solder and effectively strengthen the solder joint. Higher Ag wt. % will also enhance the dissolution and grain boundary grooving of Cu substrate, but this also imposes a side effect, i.e. it increases the melting temperature which in real life depends on the temperature limit of the substrate (Jenn *et al*., 2007a). The Bi-Ag alloy system, coupled with the optimum level of Ag wt. % with small amount of rare earth element, has a better potential as a candidate for high leaded solders. This is because the addition of a small amount of rare earth element will not influence the melting temperature and electrical conductivity of the solder joint (Yaowu Shi *et al*., 2009). Nonetheless, further research needs to be done on Bi-Ag alloys. For example, a study is required to determine the effect of doping with other elements such as tin or rare earth element on Bi-Ag solders to substitute for the lead containing alloys.

Even with some of the limitations arising from the use of Bi-Ag solders as high temperature solder candidates such as reliability and manufacturing issues, Bi-Ag alloy is still a promising material and thus, further research work in this field is required (Kim *et al*., 2002; Rettenmayr *et al*., 2005; Song *et al*., 2006; Suganuma, 2007).

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