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A high-entropy alloy as very low melting point solder for advanced electronic packaging

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ABSTRACT

SnBiInZn-based high-entropy alloy (HEA) was studied as a low reflow temperature solder with melting point around 80 °C. The wetting angle is about 52° after reflow at 100 °C for 10 min. The intermetallic compound (IMC) growth kinetics was measured to be ripening-control with a low activation energy about 18.0 kJ/mol; however, the interfacial reaction rate is very slow, leading to the formation of a very thin IMC layer. The low melting point HEA solder has potential applications in advanced electronic packaging technology, especially for biomedical devices.

1. Introduction

While Moore's law in Si chip technology is approaching to its physical and economical limit, electronic packaging technology is becoming critically important to sustain the future computational growth in microelectronics industry. The trend in miniaturization of very-large-scale-integration is moving from 2D IC to 3D integrated circuit (IC) [1–3]. The latter has various chips stacking vertically, which requires the development of new technologies such as through-Si-Via and microbumps. More importantly, the 3D IC packaging technology will need to use a hierarchy of solder joints. In other words, low (around 100 °C), middle (200 °C), and high (300 °C) wetting temperature solders will work together, so that different components can be stacked and integrated. At the moment, we have the high-Pb $Pb_{95}Sn_5$ solder for the high melting point and the eutectic SnAg solder for the middle melting point [4,5]. But for the low melting point, we only have eutectic SnBi, which has a melting point of 138 °C with a soldering temperature about 150 °C [6,7]. It would be better if we could lower the soldering temperature furthermore to 100 °C. Moreover, as the increase of electronics diversity, we are seeing the development of soft electronics, biomedical devices, solar cells, and so on. For these applications, it is important to have low reflow temperature solder applied to related packaging technologies because the working temperature of these devices is low.

How to develop industrial applicable Sn-based low melting point solders will be problematic. Eutectic binary Sn-based solders have been studied for decades; however, so far we have no appropriate solders with a melting point below 180 °C, not to say solders with a melting point below 100 °C that can be applied to biomedical devices. Because binary Sn-based solder can no longer fit itself to the fast technology development, the research on multicomponent solder is essential. Multicomponent alloys have the unique properties in the form of high-entropy alloy (HEA), especially in creep resistance [8], magnetic properties [9], biocompatibility [10], deformation behavior [11], and sluggish diffusion effect [12]. The application of HEA alloys as solders may bring these unique properties to solders, which can bring up a new area in the design of solders and broaden the applications of solders. In this article, we explore an HEA alloy as solder. The solder

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Low melting point solders based on Sn, Bi, and In elements

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A B S T R A C T

In the big data era, Si chips are integrated more and more to satisfy the fast growing demand from customers. In addition, in the post-COVID-19 virus era, the trend of distance teaching and home office has increased greatly the need of advanced consumer electronic products. To provide more processing tolerance and to build a wider temperature window in manufacturing, it becomes necessary to develop low melting temperature solders because a hierarchy of solders is needed in the packaging technology. Much research has been on Pb-free and Sn-based solders with a melting point from 180 to 230 °C. In this review, we will concentrate on low melting point solder alloys with a melting point lower than 180 °C and even below 100 °C. We review eutectic SnBi, eutectic SnIn, and the alloys with trace addition of a third element to them. Eutectic Sn-Bi solder is too brittle, and Sn-In solder is too soft. However, third element addition can only improve the solder properties partially; thus, we will further review the properties of Sn-Bi-In ternary alloys and the effect on the addition of a fourth element to them. This approach, when we include the Cu substrate, becomes a 5-element system, which leads us to consider high entropy alloys for soldering. With these Sn-, Bi-, and In-based alloys, we hope to develop new ways to design industrial applicable low melting point solders.

1. Introduction

As we enter the big data era, mobile consumer electronic products are ubiquitous. At the moment, due to the pandemic of COVID-19 virus, distance teaching and home office have increased greatly the need of advanced consumer electronic products, demanding a smaller form factor, larger memory, more function, cheaper cost, and superb reliability. Yet, at the same time, the Moore's law of miniaturization is near ending. To go to more-than-Moore and to fulfill the needs is challenging the microelectronics industry [1–3]. A promising way to sustain Moore's law is by the development of electronic packaging going from 2D IC to 3D IC, in which interposer, through-Si-via (TSV) and micro-bump are introduced to achieve the vertical stacking of chips, as shown in Fig. 1 [4]. Thus, a hierarchy of solder joints is needed, so the melting point of solder is of concern. This is because the first level of solder joints should have the highest melting point, so during the processing of the second-level solder joints, the former will not melt.

In the hierarchy, the highest melting point solder is the high-Pb solder, about 95Pb5Sn, which has a melting point just over 300 °C. The second level is the eutectic SnPb with a melting point of 183 °C or the eutectic Sn-Ag or Sn-Ag-Cu with a melting point around 217 °C. The third level is the eutectic Sn-Bi, which has a melting point of 138 °C. The high melting point of a solder has had two undesirable effects on packaging technology. First is the warpage of an interposer because the thickness of the interposer is about 50 μm which is much thinner than the typical Si wafer of 200 μm. The second is the thermal stability of a polymer substrate which tends to have a low glass transition temperature, especially when Si chips are bonded to a polymer substrate. Actually, the reflow temperature of a solder is typically 30 °C above the melting point of the solder used. Thus, in the industry it has been recommended that the maximum liquidus temperature of a solder is 225 °C and 'pasty range' ($T_{liquidus}-T_{solidus}$) is less than 30 °C [5]. As a result, the eutectic Sn-Ag and Sn-Ag-Cu became the best replacement of eutectic Pb-Sn solders [6,7].

However, recently, there is a growing demand for solders with melting point lower than 180 °C. One of the reasons is to satisfy the requirement from computational trends. When the chips become more functionalized, the packaging structures become more integrated. In this way, the chip size increases, and warpage issue

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Atomic insights of Cu nanoparticles melting and sintering behavior in Cu—Cu direct bonding

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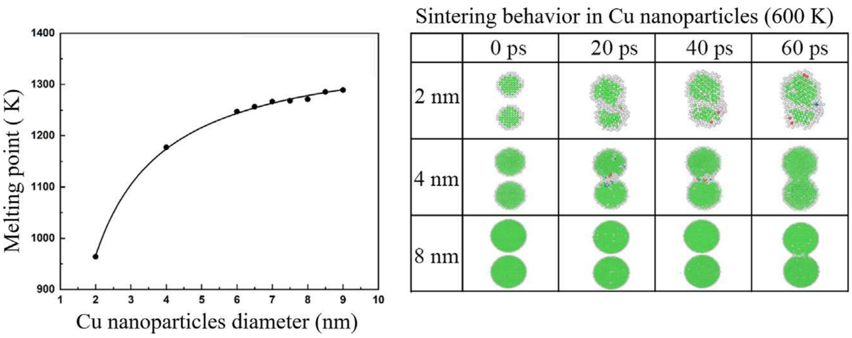
HIGHLIGHTS

- Molecular dynamics method is applied to simulate Cu nanoparticles behavior during Cu—Cu direct bonding process.
- The melting point of Cu nanoparticles from 2 nm to 9 nm ranges from 963 K to 1289 K.
- The sintering time for 2 nm nanoparticles is less than half of that for 8 nm nanoparticles.
- 2 nm nanoparticles can effectively reduce the bonding temperature and time in Cu—Cu direct bonding.

GRAPHICAL ABSTRACT



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ABSTRACT

With a layer of Cu nanoparticle slurry, it's promising to achieve fast Cu—Cu direct bonding at low temperature. To have a deeper insight and better control of the process, we apply molecular dynamics method to simulate the melting and sintering behavior of Cu nanoparticles during the direct bonding process. The melting points of nanoparticles from 2 nm to 9 nm are simulated to be from 963 K to 1298 K. The smaller the diameter of the nanoparticle, the less stable it is. At the same sintering temperature, the sintering time for 2 nm nanoparticles is less than half of that for 8 nm nanoparticles. Based on these atomic insights, if we can synthesis Cu nanoparticles as small as 2 nm, the Cu—Cu direct bonding temperature and time can be reduced further.

1. Introduction

Tin based lead-free solders have been widely applied in electronic packaging as a substitution of eutectic Sn—Pb solder for years [1]. However, together with the scaling trend in transistors, there is also a scaling trend in packaging interconnections, and the size of solder joints becomes smaller and smaller, leading to manufacturing challenges and reliability concerns in lead-free solder technologies [2,3]. Cu—Cu direct bonding has been studied as a replacement for solder in the future ultra-high-density interconnect technologies. Compared with the traditional solder joints, Cu—Cu direct interconnection has better scalability, better conductivity, thermal conductivity, and resistance to electromigration [4]. However, because the Cu surface is easy to be oxidized and the melting point of copper is high compared with Sn, Cu direct bonding is always achieved under very strict environment, i.e. the bonding temperature is usually above 300 °C, and it needs to be bonded in ultra-high vacuum chamber [5–7]. Nanoparticles have been studied to be applied in Cu—Cu direct bonding in order to decrease the bonding temperature and prevent surface oxidation. Li [8] et al. prepared Cu nanoparticles slurry for Cu—Cu interconnection. The diameter of Cu nanoparticles is distributed in the range of 80–120 nm, and the bonding temperature can be reduced to 250 °C, with 30 min of heating and 30 min of annealing. Kim [9] et al. synthesized Cu nanoparticles with

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Ultra-thin intermetallic compound formation in microbump technology by the control of a low Zn concentration in solder

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ABSTRACT

We report here the extremely slow intermetallic compound (IMC) growth kinetics in the reflow reaction between a Sn-based solder of SnBiIn-2 at.% Zn and Cu. The solder has a melting point about 90 °C, and after reflow for 5 min on Cu at 120 °C, the formed IMC was Cu_5Zn_8 with a thickness only about 0.36 μm, which is much thinner than the IMC in nowaday packaging technologies. We systematically studied the IMC growth kinetics and built up a model to explain the extremely slow IMC growth rate. The growth kinetics of the reaction is non-parabolic and the activation energy is about 23.8 ± 1.6 kJ/mol. The non-parabolic kinetics is related to the lateral grain growth in IMC during the reactive diffusion along the moving grain boundaries. Our theoretical model shows that the growth rate of Cu_5Zn_8 compound should be proportional to the square root of Zn initial concentration in solder and a low Zn concentration in the solder will lead to a very slow IMC growth rate. The finding could be applied to control IMC thickness in 3D integrated circuit (3D IC) with micro-bump technology.

1. Introduction

As Moore's law of miniaturization in Si technology is approaching its physical and economic limit, 3D IC has been regarded as the most promising technology to sustain the law in the future [1–3]. 3D IC is achieved by stacking multiple chips using TSV (through-Si-via) and microbumps. There are three different size solder joints in the 3D architecture, including Ball Grid Array (about 760–200 μm), Controlled Collapse Chip Connection (C-4 joints about 100 μm) and microbumps (about 20 μm). In the future, the density of input/output connections in packaging will increase, so the size of μ-bump might be scaled down to 10 μm, 5 μm, or even only 1 μm [4–6]. This scaling trend will lead to serious reliability concerns and challenges in microbumps.

One of the main challenges is to control IMC thickness in microbumps. This is because the diameter of microbumps has been reduced more than 10 times from the C-4 joint, so the volume of solder will be reduced more than 1000 times [7]. Under the same reflow time, if we assume both the traditional solder balls and microbumps have the same IMC growth rate, the percentage of IMC would be much higher in microbumps. Furthermore, the IMC growth rate in small size solder bumps will be remarkably higher due to surface diffusion during interfacial reaction [8]. Actually, the solder layer in microbumps could transform completely into IMC after just aging for 24 h at 180 °C [9]. IMC is brittle in nature, and the high percentage of IMC will lead to the embrittlement problem in microbumps [10]. In addition, during IMC growth, solder layer will be experiencing volume shrinkage, and volume shrinkage works together with electromigration would lead to early failures [11–13]. Therefore, it's essential to control the IMC growth rate in the interfacial reaction between the solder and under bump metallization (UBM) in microbumps.

In this study, we report a Sn-Zn solder containing very low concentration of Zn solder that has an extremely slow reaction rate with Cu substrate. Some researchers have already investigated the effect of adding Zn to Sn-based solder to slow down the IMC growth kinetics [14–19]. However, the IMC growth rate in our work is much slower than the published results. Moreover, the reason of Zn effect to IMC growth kinetics is not systematically explained in those works, because Sn–Zn–Cu is a ternary system, and the reaction paths are complicated. Therefore, we developed a theoretical model for a systematic discussion of the competition among evolution paths in reactions between Cu with Sn-Zn solder. We explained that only a small amount of Zn can lead to the extremely slow reaction rate in IMC formation. The finding is important in the application of microbumps to advanced electronic packaging technology.

2. Experimental

SnBiIn-2 at.% Zn solder were prepared using high purity (>99.9%) Sn, Bi, In and Zn according to atomic ratio of Sn:Bi:In:Zn = 48:25:25:2. The ingots melted completely at around 300 °C in a vacuum induc-

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Effect of adding Ag to the medium entropy SnBiIn alloy on intermetallic compound formation

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ABSTRACT

Wetting reactions of the medium entropy alloy of SnBiIn and SnBiInAg on Cu substrate have been investigated. The melting points of both SnBiIn and SnBiInAg are about 80 °C. The reactions were performed at 120 °C, 140 °C, and 160 °C. The kinetics of interfacial intermetallic compound (IMC) growth of Cu_6Sn_5 was studied to be ripening-controlled with activation energy about 11.1 kJ/mol for SnBiIn and 10.4 kJ/mol for SnBiInAg. However, the addition of Ag has effectively reduced the IMC growth by 20–30%. We propose that it is because Ag has reduced the pre-factor of diffusivity by increasing the entropy of the alloy.

1. Introduction

As we enter the big data era, the trend in very-large-scale-integration (VLSI) of Si technology is moving from 2D IC to 3D IC. At the same time, the challenge in integration of electronic materials has been greatly increased. Take the example of solder micro-bump, while the diameter has been reduced more than 10 times from the flip chip C-4 joint, the volume of solder is reduced more than 1000 times [1–4]. How to control IMC growth and limit the percentage of IMC in micro-bump is critical, so new solder materials are under consideration. High entropy alloy (HEA) is relatively new, first reported in 2004, and it has attracted attention for low temperature soldering applications [5–7]. The possible sluggish diffusion kinetics in some HEAs might reduce the brittle interfacial IMC growth in solder joints [8]. So far, there are few reports about HEA solder. We note that according to the phase diagram of SnBiIn ternary alloy, there is a stable Sn-rich solid solution, which can react with Cu to form solder joints [9]. For comparison, we add 10% Ag to SnBiIn in order to increase its mixing entropy. The effect of adding Ag to SnBiIn on reducing IMC formation is reported here.

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2. Experimental

The SnBiIn medium entropy solder was prepared using high purity (>99.9%) Sn, Bi and In according to atomic ratio of Sn:Bi: In = 42:28:30 in a vacuum induction furnace in Ar atmosphere. The SnBiInAg alloy was prepared by adding 10 atomic % of Ag into SnBiIn alloy. Tiny pieces about 5–10 mg were cut from the solder bulk for differential thermal analysis (DTA) to determine the melting point of the two solders. The wetting samples on Cu were placed on hot plate and reflowed at 120 °C, 140 °C and 160 °C for 5 min, 10 min and 20 min, respectively. The cross-sections of wetting samples were investigated by scanning electron microscope (SEM) and energy dispersive X-ray spectroscope (EDX). The interfacial IMC thickness was obtained from the SEM images by the software ImageJ.

3. Results and discussion

In order to identify the melting behavior of the SnBiIn and SnBiInAg alloys during reflow, the SnBiIn and SnBiInAg alloys were analyzed using DTA. As shown in Fig. 1(a) and (b), the melting points of both SnBiIn and SnBiInAg are about 80 °C. The addition of Ag seems to have little influence on the melting point. However, the melting peak in the DTA curve of SnBiInAg solder joints is

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Surface diffusion controlled reaction in small size microbumps

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ABSTRACT

We made head-shaped Cu-Sn solder joints with diameters about 10 µm, 20 µm, and 50 µm and reflowed the samples at 240 °C, 260 °C, and 280 °C for 60 s to 600 s. In the 10 µm bumps, there is only one Cu_6Sn_5 grain after reflow, thus the classic model of scallop-type grain growth of Cu_6Sn_5 does not apply. Also, the grain growth in the small size bumps has the faster growth rate. We proposed a surface diffusion-controlled model to explain the new kinetics. According to our model, the diffusion activation energy is calculated to be Q_S = 0.18 ± 0.02 eV/atom and diffusion frequency factor to be D_{S0} = 5.65 × 10^{-3} cm^2/s.

1. Introduction

Similar to the scaling trend in chip technology, there is a scaling trend in packaging technology. The size of solder joint shrinks from several hundred microns to about 20 µm and may eventually shrink to several microns with the development of packaging technology [1–3]. In the traditional flip chip C-4 solder joints, surface diffusion is neglected compared to grain boundary diffusion and lattice diffusion, because the surface to volume ratio is small. However, when the bump size decreases to less than 20 µm, surface diffusion becomes dominant. While surface diffusion in solid state solder joint reactions has been studied [4], surface diffusion in reflow reactions hasn't been analyzed. In this work, we study the surface diffusion-controlled kinetics of wetting reaction in solder joint formation.

2. Experimental section

To fabricate the head-shaped Cu-Sn solder joints, we made arrays of holes with diameter of 10 µm, 20 µm, and 50 µm, in the photoresist by lithography. Then, Cu layer with a thickness of 4.5 µm was electroplated in the holes, followed by electroplating of 6.5 µm Sn layer. After cleaning the photoresist away, the Cu-Sn bumps were reflowed for different length of time in a reflow oven under N_2 environment after Ar^+ plasma pretreatment. We placed the chips up-side down to avoid the influence of gravity on surface diffusion. The samples after reflow were polished and we observed the cross-sections by scanning electron microscope (SEM). For each bump size, we pick three bumps out of the array and measured the thickness of IMC by Image J.

3. Results and discussion

Fig. 1 shows the SEM cross-sectional image of the bumps after being reflowed. The grain size in the 50 µm bumps is about 2–3 µm after reflowed for 1 min and about 10 µm after reflowed for 10 min. Under the same reflow time, the 20 µm bumps have bigger grains than 50 µm bumps. This trend is especially much more prominent in 10 µm bumps. Almost all the 10 µm bumps start to have only one grain, which is about 10 µm in diameter within 1 min reflow. The above finding that IMC has the fastest growth rate in the smallest bump is unexpected from previous studies [5,6].

To study the IMC grain size in the bumps, we etched the unreacted solder away and Fig. 2(a) shows the after etching image for the 20 µm bumps after reflow at 240 °C for 1 min. In Figs. 1 and 2, the complete and incomplete wetting of grain boundaries by the melt phase can be observed, where the Sn-based liquid phase during the soldering fully or almost fully separates the grains of the underlying Cu_6Sn_5 phase [5]. The grain boundary wetting transitions can influence the grain growth behavior observed in Figs. 1

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国家自然科学基金资助项目批准通知

刘影夏　先生/女士：

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2. **2019年9月18日16点：**提交电子修改版计划书的截止时间;
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附件：项目评审意见及修改意见表

国家自然科学基金委员会

2019年8月16日

附件：项目评审意见及修改意见表

项目批准号	51901022	项目负责人	刘影夏	申请代码1	E010702
项目名称	应用于先进电子封装的低熔点高熵合金焊料焊接性与可靠性研究				
资助类别	青年科学基金项目		亚类说明		
附注说明					
依托单位	北京理工大学				
直接费用	25.00 万元		起止年月	2020年01月 至 2022年12月	

通讯评审意见:

<1>具体评价意见:

一、请针对创新点详细评述申请项目的创新性、科学价值以及对相关领域的潜在影响。
项目拟研制高熵合金焊料解决低温度下集成电路的封装问题，既有创新性又有应用的迫切性。研发的新材料对解决目前封装所需的低温焊料具有重要意义。

二、请结合申请项目的研究方案与申请人的研究基础评述项目的可行性。
研究方案可行，前期有很好的工作基础，材料体系基本确定，在本项目中进行组分优化、性能测设等工作。申请人与领域前沿科学家有很好的合作关系，有利于项目的开展。优先支持。

三、其他建议

<2>具体评价意见:

一、请针对创新点详细评述申请项目的创新性、科学价值以及对相关领域的潜在影响。
申请人拟通过研究不同组分的高熵合金与熔点之间的关系，研究其焊接性能，并研究高熵合金对金属间化合物反应动力学的影响，筛选出适合封装工业用的低温钎料。虽然，工程上钎料合金的成分趋于简单，较多组元成分的钎料合金由于工艺过程中容易变化，从而不受工业界欢迎。该项目研究工作值得尝试研究。

二、请结合申请项目的研究方案与申请人的研究基础评述项目的可行性。
申请者前期有一定的研究基础，项目具有一定的可行性。

三、其他建议
无。

<3>具体评价意见:

一、请针对创新点详细评述申请项目的创新性、科学价值以及对相关领域的潜在影响。
芯片封装技术正通过复杂化、高集成化来满足消费者对于电子产品性能日益苛刻的要求。该项目提出采用低熔点高熵合金作为低温焊料，降低回流焊接时的温度来解决封装焊点小型化过程中微凸点的金属间化合物生长动力学控制以及大尺寸封装翘曲度等问题，具有较好的创新性。通过研究不同组分的高熵合金和熔点之间的关系，探寻高熵合金组分和结构及其熔点和焊接性能之间的科学关联，筛选出适用于封装工业焊接用的低熔点高熵合金焊料，有利于降低传统焊料熔点难题，为先进封装所面临的问题和挑战提供一种解决途径，具有较高的学术价值和科学价值。同时，将回流焊接技术应用于制备低温焊料，改良助焊剂，高熵低温焊料利于克服未来电子封装小型化、复杂化所带来的挑战，具有一定的实际应用意义。

二、请结合申请项目的研究方案与申请人的研究基础评述项目的可行性。
项目的研究内容较新颖，研究方案可行性高、具体完整，项目组的实验设备完善，申请者具有较好的研究基础。

三、其他建议
建议基金委考虑对本项目予以资助。

修改意见:

工程与材料科学部

2019年8月16日