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Beyond Lead: Exploring Fillers and Reinforcements in Lead-Free Solder Technology

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Abstract. Due to tighter restrictions on the use of hazardous lead-bearing solder alloys, lead-free solder research has seen significant growth in recent years. The most common SAC alloys have emerged as viable candidates for the substitution of conventional Sn-Pb alloys among the representative lead-free solders (Sn-Cu, Sn-Bi, Sn-Zn, Sn-In, Sn-Ag, and Sn-Ag-Cu (SAC)). These alloys have limited use in contemporary microelectronic packaging devices due to various worries about the existence of brittle intermetallic compounds (IMCs) like Ag_3Sn and Cu_6Sn_5 in these materials. Over the years, numerous lead-free solder alloy alternatives with nanoparticle reinforcement have been proposed as an alternative to limit the growth of IMCs and enhance solder joint durability. This paper details the development of lead-free solders with selected fillers and reinforcements to date. The thermal cyclic test method was also discussed as one of the alternatives for reliability test techniques to be explored in future studies on this topic. In conclusion, fillers and reinforcements are also essential for enhancing interconnection's heat cycle efficiency. Exploring various fillers and reinforcements that can be used to advance lead-free solder technology with thermal cyclic methods will open more investigation opportunities for lead-free solders.

Introduction

Electronic devices have advanced toward downsizing and multifunctionality due to the quick development of electronic packaging technology [1,2]. This is especially true for electronics used in wearable technology, smart watches, smart phones, and cutting-edge cars [3]. The number of solder joints has dramatically grown even if the size of interconnections has been drastically reduced. Since they offer both the mechanical and electrical connection between the various levels of the package, solder joints are essential to electronic packaging [4, 5]. Warpage and solder joint fatigue life would be the two most important reliability problems associated with downsizing [3]. Thermal expansion mismatch between the materials was the primary source of these two problems [3, 4, 5]. In the microelectronics industry, thermal cycle tests and fatigue tests are two of the most popular reliability test techniques. The main goal of a fatigue test is to evaluate the mechanical toughness of solder junctions under cyclic mechanical loads. It imitates the mechanical strains that solder junctions endure throughout the operating life of electronic equipment as a result of things like heat expansion and contraction, vibration, and mechanical shocks. Solder junctions are typically the weakest and most prone to fatigue due to the low strength and elastic modulus of solder materials [6]. Overall, two distinct techniques are employed to assess the dependability of lead-free solder joints: heat cycling testing and fatigue tests. Thermal cycling testing concentrates on how temperature variations affect the solder junctions, whereas fatigue tests concentrate on how mechanical stresses affect them. Both kinds of tests can offer insightful data regarding the functionality and dependability of lead-free solder joints.

Since reliability is a key issue in solder joint, there are many approaches to enhance the reliability of the solder joint. One method is by adding additional material in small amount to the solder material in micro or nano size. These additional materials are usually referred to as reinforcement if the major side effect is to strengthen the solder joint. Reinforcements can include various forms of particles or fibers, such as micro-sized particles of ceramic materials (e.g., alumina), metallic particles (e.g., nickel), or even carbon nanotubes [7, 8]. Another type is fillers. Fillers are materials that are added to the solder material to modify its properties, such as its melting temperature, wetting characteristics, mechanical strength, and thermal conductivity. Common filler materials include bismuth, indium, antimony, and other elements or alloys that can be added in small quantities to the main solder composition to affect its characteristics such as thermal expansion, mechanical strength, and solder joint reliability [9]. Basically, the main difference between fillers and reinforcements in lead-free solders lies in their purpose and the effects they have on the solder's properties. Fillers are primarily used to modify the solder alloy's characteristics, such as melting temperature and wetting behavior. Reinforcements, on the other hand, are added to enhance the mechanical strength and durability of solder joints, making them better suited to withstand mechanical stresses and other external forces. Both fillers and reinforcements contribute to the development of lead-free solder materials that meet the demands of modern electronics, offering improved performance, reliability, and sustainability in the face of miniaturization and multifunctionality. However, to date, there are limited studies available in the literature on thermal cyclic and fatigue on lead-free solder interconnections with reinforcements. Most of the studies focus on other mechanical study such as tensile, shear, and hardness impact on the lead-free solder from the addition of fillers and reinforcement.

Lead-Free Solders with Fillers and Reinforcement

Pb cannot be used in electronic components anymore due to its toxicity, hence lead-free solder materials were investigated as alternatives. These lead-free solders still fall short of the actual manufacturing requirements to replace leaded solders due to their high melting point, poor wettability and dependability, and high cost. Therefore, it is critical to find a practical method for enhancing the performance of solder alloys. The major methods being used by researchers right now to enhance the performance of lead-free solders are microalloying and strengthening micro- or nanoparticles with the inclusion of fillers and reinforcements [9, 10].

Microalloying is a method for improving the properties of solder by adding traces of alloying elements to change the composition of the solder. The two main groups into which the alloying elements can be divided are rare earth (RE) elements (Ce, La, Pr, Nd, etc.) and other metal elements (Ga, In, Mg, Ni, Ag, etc.) [11, 12]. This method still has a lot of issues that keep it from being extensively used with lead-free solders that have little alloying elements. For instance, too many RE components may result in the growth of Sn whiskers on the surface of solder connections, which will cause an electrical device to fail sooner than expected [15]. While lead-free solder's wettability, mechanical properties, and grain refinement are significantly impacted by strengthening micro and nanoparticles [16, 17]. Thus, a growing number of researchers are concentrating on the production and performance characterization of composite solders boosted by various particles. The micro and nanoparticle kinds that have received the greatest attention to date include metals (Cu, Ni, Mn, Co), oxides (La_2O_3, Fe_2O_3, TiO_2, etc.), ceramics (SiC, TiC), and carbon materials (carbon nanotubes (CNTs), graphene nanosheets (GNSs), etc.) [9, 10]. The strengthening impact of micro and nanoparticles still comes with several issues or unknown effects, despite the fact that much research has shown the positive impact of filler addition. For instance, it is not apparent how particles affect the melting characteristics of lead-free solder [18,19]. Additionally, composite solders enhanced by micro and nanoparticles are not used in the electronic industries due to their high cost and labor-intensive preparation. In order to follow the trend of environmentally friendly development and to satisfy the demands of the electronics industry for packaging materials, the impact of micro and nanoparticle doping on the microstructure and properties of lead-free solders need to be thoroughly examined. The analysis is anticipated to serve as a theoretical guide for the creation of high-performance micro or nanoparticle-reinforced lead-free solders.

In order to understand the effect of additional fillers and reinforcement material, there are several aspects of research to be studied. One is microstructure. This is to study agglomeration of the added element or particles which would be a concern for initial sites for formations of cracks or voids. Another aspect to be studied under microstructure is the formation of intermetallic compounds also known as IMCs. IMCs is a brittle layer formed at the interface between the molten solder and substrate. It can also form in the solder bulk. Since it is brittle in nature, an overgrown amount of this phase would reduce the solder joint reliability. The addition of microalloying and micro or nano particles was expected to influence IMCs formation be it changing into a different type of IMCs or affecting the rate of its formation. Fig 1 shows an example of IMCs formation between SnAgCu solder on Ni-P surface finish.

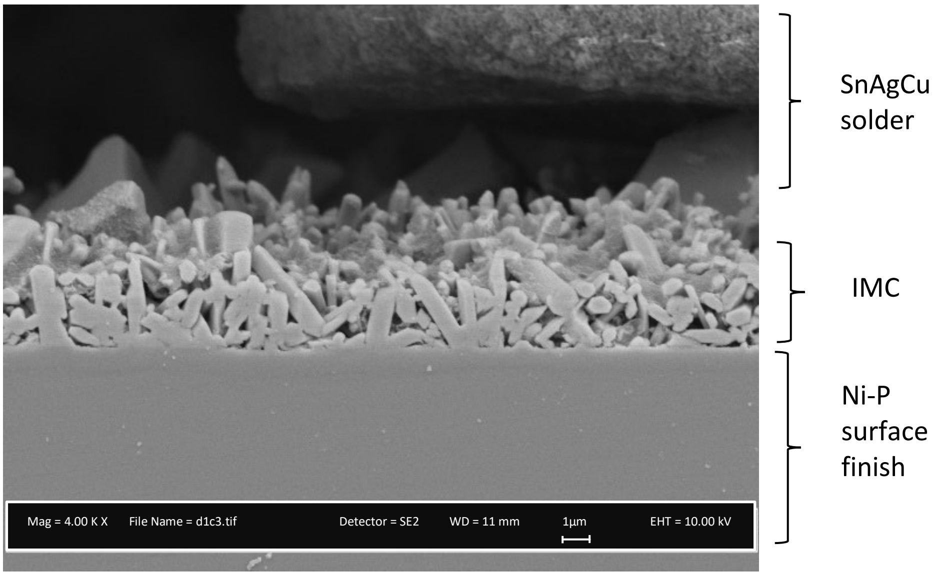

Fig 1. IMCs formation for as reflow sample of Sn4.0Ag0.5Cu on Ni-P electroless coated Cu substrate.

The mechanical properties are the next topic of research. Tensile properties, shear strength, and hardness values can be quantified for a direct measurement of the impact of microstructure modification. These are the basic mechanical tests that must be performed on solder joints in accordance with particular specifications because solder junctions need to be strong enough to withstand the buildup of residual stress brought on by CTE mismatches during operation [20]. Wettability is a further crucial factor. This is done to see how the molten solder spreads out on the substrate. The surface energy would change with the addition of fillers and reinforcing particles, affecting the solder's wettability. Adding graphene nanosheets (GNSs) to SnAgCu solder is one example [21]. The study demonstrates that adding GNSs at the right concentration can greatly increase the spreading area of SnAgCu solder. However, when the concentration is higher than 0.1 weight percent, GNSs have a tendency to aggregate and float on top of the molten solder, which reduces the composite solder's wettability [21].

Mechanical shocks, vibrations, and temperature cycling are the three main conditions that might result in solder failure in a semiconductor package [3]. The reliability of the product as a whole is significantly influenced by these three variables. Therefore, reliability testing would be another crucial characteristic. The solder connection was heated during reliability testing in ways that either mimicked the manufacturing process, such as numerous reflows, or the working environment, like isothermal aging and thermal cyclic tests. This is related to the IMCs compound growth as it is exposed to heat. The aim of these tests would be to accelerate the growth or evolution of IMCs at the solder joint so that the mechanical test can be conducted on the solder joint to predict the joint reliability and feasibility of the micro-alloy or particles in improving the solder joint reliability. Other than accelerated thermal exposure, accelerated mechanical stress exposure such as fatigue test would also be useful in predicting the solder joint life. Another alternative is to evaluate the effects of thermal cycling and external additional loads at the same time, hence, the solder joints undergo thermomechanical coupled cyclic loading. Or focusing on the low load effect on reliability over a long period of time, such as the indentation creep test [6, 9]. However, the challenge with these tests would be the extended time required for the test to complete.

Another test that might be performed is a corrosion test, which would assess the impact of an extra element, particularly for solder that is vulnerable to corrosion, as Sn-Zn solders [22]. Sn-Zn solders have a number of intriguing characteristics, including a low price and a 220°C reflow temperature. Additionally, Sn-9Zn eutectic alloy offers higher mechanical qualities than traditional Sn-Pb solders and has a melting temperature (198°C) that is comparable to Sn-Pb eutectic alloy's (183°C) [23,24]. While some Sn-Zn composition regions can function as sacrificed anodes with good anticorrosive properties for the ferrous base metals, the Zn in the Sn-Zn system is easily oxidized to form certain corrosion products (e.g., Zn oxide/hydroxide and $ZnCl_2$) that reduce the reliability of solder joints and wettability [25,26]. This has led to a lot of attention being paid to the study being done to see if it's possible to create Sn-Zn lead-free solders. As a result, numerous Sn-Zn alloy systems have been created with various alloying elements, including Ag, In, Cu, Zn, Bi, and Sb. Their microstructures, mechanical characteristics, and solderability have also been described [25-28].

Conductivity tests are another sort of test, and an example of their use is to examine the impact of adding ceramic particles to lead-free solders on the conductivity value of the solder. The purpose of an electromigration test is to determine how electrical flow affects the development and evolution of IMCs over time. While the drop test is a technique used to assess the dependability of solder junctions after exposure to impact force, it is mostly utilized in handheld electronic devices. The sample is dropped from a certain height during a drop test analysis in order to get the necessary acceleration. The results of the drop test can be used to evaluate the stresses experienced by the solder balls either experimentally or using a simulation model [3]. Table 1 shows examples of solder joint properties with the addition of nanoparticles.

Table 1. The mechanical characteristics of lead-free solders enhanced with nanoparticles [9].

Solders	Ultimate Tensile Strength (MPa)	Elongation (%)	Microhardness
Sn3.0Ag0.5Cu	46.0	29.5	-
Sn0.3Ag-0.5Cu/0.1Mn	56.5	-	-
Sn3.5Ag0.25Cu/1TiO_2	70.1	25.2	18.5 HV
Sn3.0Ag0.5Cu/0.05La_2O_3	79.0	14.1	13 HV
Sn3.0Ag0.5Cu/0.03GNSs	50.3	23.5	-
3Sn3.0Ag0.5Cu/0.1GNSs	50.7	21.5	-
Sn3.0Ag0.5Cu/0.2Ni-GNSs	58.4	-	14.6 HV
Sn3.0Ag0.5Cu/0.05Ag-GNSs	50.1	14.7	-
Sn3.8Ag0.7Cu/1CNTs	56.7	24.4	-
Sn58Bi	81	21	0.30 GPa
Sn58Bi/0.01GNSs	84	32	0.26 GPa
Sn58Bi/0.1GNSs	93	17	0.41 GPa
Sn58Bi/0.03CNTs	94.2	21.7	-
Sn9Zn	41.0	43.4	13.6 BHN
Sn9Zn/1.0Ag	43.6	19.4	16 BHN
Sn9Zn/1.0Sb	53.6	21.6	15.9 BHN
Sn9Zn/1.0Al_2O_3	43.4	44.9	27 BHN

The results of adding nanoparticles to lead-free solders are shown in Table 1 above. Generally, the trend indicates that adding nanoparticles enhances the solders' tensile strength while also reducing their elongation due to their severe restriction influence on dislocation movements. A thorough assessment of mechanical characteristics and other performance metrics, including wettability, reliability testing under heat and/or stress, corrosion value, conductivity, and others, is therefore necessary to identify the optimal composition. This leads to our next subject, the thermal cyclic test.

A style of reliability test that investigates how the solder junction is affected by thermal exposure cycles. The section below will explain how the test is conducted and the type of results that can be obtained.

Thermal Cyclic Test

A thermal cyclic test for lead-free solder is a laboratory procedure designed to assess the reliability and durability of solder joints under repeated temperature changes. This type of test is particularly relevant for electronic components, circuit boards, and assemblies, where temperature fluctuations due to operational conditions can impact the solder connections over time. During the thermal cyclic test, a sample or specimen containing lead-free solder joints is subjected to alternating cycles of temperature extremes. These extremes typically include exposure to high and low temperatures, simulating the real-world conditions that electronic devices may encounter during their operational lifespan.

The thermal cyclic test provides insights into how lead-free solder joints perform under conditions of thermal expansion and contraction, which can lead to mechanical stress on the joints. Since a larger coefficient of thermal expansion (CTE) mismatch exists between the chip (CTE_{Si} = 2.6 × 10^{-6}/K [29]) and solder (CTE_{SAC305} = 23 × 10^{-6}/K [30]) compared with what is between the solder and the PCB substrate (CTE_{FR-4} = 18 × 10^{-6}/K [30]), resulting in a tense strain field accumulated between the chip and the solder during thermal cycling [31].

The test helps assess the long-term reliability of soldered connections and can inform design decisions, material choices, and manufacturing processes to enhance the durability of electronic assemblies. Ultimately, the goal of conducting a thermal cyclic test for lead-free solder is to ensure that solder joints remain robust and functional even when exposed to the temperature variations that can occur during the use of electronic devices. This contributes to the overall quality, reliability, and performance of electronic products in various industries. Figure 2 show an example of how the sample and the thermal cycle sample looks like. Yihui Du et al. [31] used Sn3.0Ag0.5Cu solder. The package size is 12 × 12 × 1 mm^3 (hollow 22 × 22 Au/Ni/Cu pads with 0.28 mm in diameters at 0.5 mm pitch) as can be seen in Fig. 2 (a) and (b). The temperature cycles is between − 55 °C to 125 °C for 2500 cycles. The main purpose of the study was to analyze recrystallization of the grains and how it effects the reliability of the solder joint. Post 2500 thermal cycles, the samples were characterized using electron backscattered diffraction technique and transmission electron microscope to study the microstructure of the cross sections.

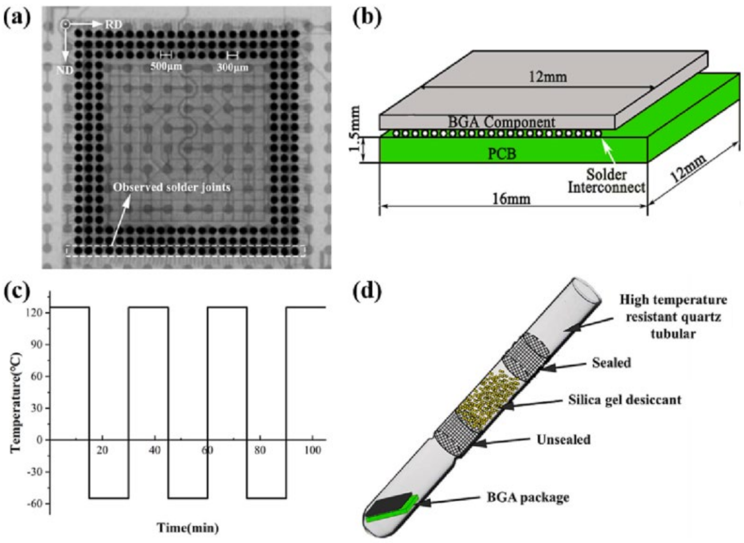

Fig. 2. Sn3Ag0.5Cu BGA packages and thermal cycling treatment (a) X-ray transmission image of the BGA packages; (b) schematic of a cross-sectioned sample; (c) temperature profile of thermal cycling; (d) schematic of the vacuum sealing [31].

Figure 3 shows an example of microstructure analysis for thermal cyclic tests. In this study, electrical reading was collected simultaneously as the sample was exposed to the thermal cycles [32]. The cyclic test was stopped upon electrical failure detection. The samples were then taken out and characterized under the optical microscope. Cracks and failure sites were then studied from the images obtained. The analyses from the microstructure image would usually relate the formation of cracks to the formation and evolution of the IMCs, and changes in the grain size of the solder materials. However, as mentioned previously, not much study has been conducted on thermal cyclic tests on lead-free solder with fillers and reinforcement. Equipment limitations and the extended time required to complete the test are the two main reasons behind this trend. Exploration on the lead-free solder with fillers and reinforcement under thermal cyclic conditions remains a largely untapped area of research. This presents a significant opportunity for future studies. Conducting thermal cyclic tests is crucial for proving the concept and ensuring the reliability of the solder composite before it can be used in the industry. Therefore, despite the challenges, it is an avenue that warrants further investigation.

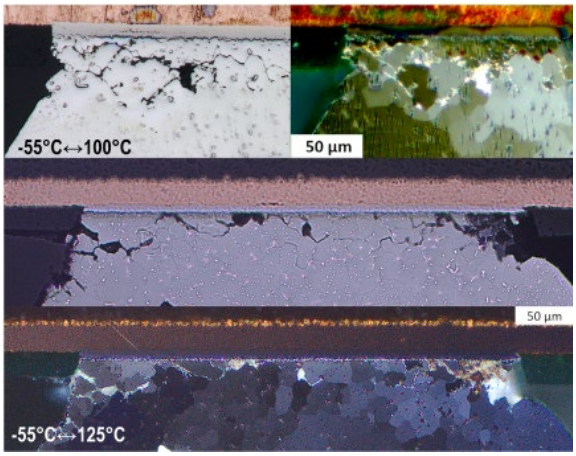

Fig. 3. Recrystallization and precipitate coarsening assisted failure of solder joints in thermal cycling, − 55°C↔100°C and − 55°C↔125°C, white and polarized light observation after electrical failure detection [32].

Conclusion

Making solder joints for electronic equipment more reliable while maintaining acceptable reliabilities is made more difficult by miniaturization and multifunctionality. One technique used to increase the solder joint reliability, especially with the changeover to lead-free soldering, is the use of fillers and micro or nano-reinforcement. One of the crucial elements of analysis is IMCs. The additional materials introduced may influence in reduction or alteration of the growth and evolution of IMCs. Various characterizations were often done on the solder joint in order to assess and optimize the amount of fillers and micro or nano particles applied. Characterization involved can be categorize into microstructure analysis, mechanical properties, corrosion resistance, conductivity, electromigration, reliability test and others. Reliability test can be divided into effect from exposure to thermal fluctuations and effect from additional loads such as vibration or shock from being dropped. The test can be carried out separately or together. Thermal cyclic test is focusing on the effect from CTE mismatched between all different type of materials in the electronic packaging when it is being repeatedly exposed to low and high temperature in cycles. Samples that went through thermal cyclic test would be analysed through visual inspection for signs of cracks, and delamination. In some cases, electrical resistance measurement was conducted to quantify the effect. All of these effects were then relate to the microstructure changes that the solder joint goes through as it goes through the cycles. Overall, thermal cyclic test is an important aspect of reliability quantification for solder joint that can be further explored for lead-free with fillers and reinforcements, and support from other characterizations is also important to enable a full analysis of the effect from filler and micro or nanocomposite addition into the solder material.

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