Cryorolling of SAC305 solder: Microstructure analysis and shear strength of solder joint

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Abstract. Miniaturization of devices along with demand of higher performance in electronic packaging industries led to development of higher reliability solder alloys. Solder material must meet the requirements of high solder joint strength, better wettability, high fatigue and creep resistance in order to provide highly reliable solder joint. This work attempts to evaluate effectiveness of cryorolling in providing ultrafine grains which could lead to higher solder strength. The microstructure of bulk solder and IMC layer at interface between solder and Cu substrate, and lap joint shear strength of cryorolled commercial SAC305 solder alloy are presented here. Solder alloys were dipped in liquid nitrogen at different dipping times (0, 10, 30 and 60 minutes) prior to rolling. Cryorolling was observed to encourage grain refinement in solder with 30 min dipping gave lowest grain size of 5.88 µm, compared to 11 µm for as-cast solder. Longer dipping up to 60 minutes resulted in larger grain size and tend to be brittle with small cracks started to appear during rolling. Shear strength test showed an improved and stronger joint for SAC305 dipped in liquid nitrogen for 30 minutes compared to sample without dipping in liquid nitrogen. As a conclusion, cryorolled solder alloys had smaller grain size and this led to an improved solder joint shear strength.

1.0 Introduction

The mechanical properties and reliability of solder joints are affected by microstructures, movement of dislocations and grains reconfiguration. As solders typically have relatively low melting point, the deformation mechanisms at either room temperature or service temperature are considered as high-temperature mechanisms. These mechanisms are dependent on microstructure, which is related to composition and soldering process. Thus, understanding the relationship between structure - property and structure – process are important to produce desired microstructure which give high reliability products [1]. For instance, larger grain size microstructure will become more resistant towards creep when grain-boundary sliding dominates the total creep strain. On the other hand, fine grain solder joint have larger fatigue life compared to coarse grain solder joint.

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Grain refinement in solder alloy is typically because of alloying [2, 3] or via severe deformation process (SPD) such as equal channel angular pressing (ECAP) [4, 5]. Although alloying is more common in refinement of solder alloy, SPD technique has been reported to be effective in production of materials with submicron or nano-sized grains [6]. One of the main advantages of SPD is that ultra-high plastic deformation (with strain in order of 10 and higher) is achieved without a substantial change in dimensions of the worked sample, thus allowing very strong grain refinement effect [7]. By combining cryogenic treatment and deformation process, the tensile strength and yield strength can be considerably improved as dynamic recovery is suppressed during cryorolling [8]. The cryogenic treatment is able to suppress the recrystallization along the process and thus preserving the grain refinement effect. Researches based on cryogenic treatment had been performed on magnesium alloy, titanium alloy and some steels, and the properties of these materials have been reported to improve to various extent [8]. Therefore, this study was developed to investigate the effect of various dipping time in liquid nitrogen prior to rolling on the microstructure and solder joint strength of commercial SAC305 solder alloy.

2.0 Methodology

A commercial Sn-3.0Ag-0.5Cu (SAC 305) solder alloys was used in this project, provided by RedRing Solder. The solder was initially prepared by melting the ingot bar and casted into stainless steel mold to get flat sheet with approximately 4 mm thickness. Several thickness reduction percentages were tried to observe deformation behavior after nitrogen dipping and it was found that 75% thickness reduction was maximum before the solders formed severe cracks and break. Dipping time was set to be 10, 30 and 60 minutes prior to rolling process.

The solder alloys after cryorolling were ground with SiC abrasive paper grit 360 until 2000 before polished using 1 and 3 μm diamond suspension. Once mirror-like surface was obtained, the samples were chemically etched with etchant of 2% hydrochloric acid (HCl), 5% nitric acid (HNO3) and 93% ethanol (CH3OH) for 15 seconds to reveal the microstructure of the alloy using ZEISS SUPRA 35VP scanning electron microscope (SEM) equipped with energy dispersive X-ray (EDX). For evaluation of shear strength of solder joint, solder samples were reflowed in T200C+ lead-free reflow oven at 280°C for 15 seconds on bare copper substrate. For all reflowed solder joints, activated rosin (RA) flux was used. Single lap joint shear test was carried out following ASTM D 1002 standard, and sample dimension is shown in figure 1. The test is conducted on Instron Universal Testing Machine at displacement rate of 13 mm/min. The maximum load by each sample was then divided by the overlap area, which is 9 mm2 to get the strength value, and the strength value presented was an average of 5 tests.

Fig.1. Sample dimension and set up for single lap joint shear test
3.0 Results and Discussion

3.1 Microstructure

Fig. 2 shows the evolution of microstructure as dipping time in nitrogen is varied, from as-cast solder to 60 minutes dipping time. All samples show typical SAC305 features which consists of a matrix of β-Sn and eutectic phase of Cu₆Sn₅ and Ag₅Sn IMC particles [9]. EDX analysis (not presented here) was done to confirm elemental composition of the IMCs. It can be observed that dipping in nitrogen encouraged grain refinement of the β-Sn matrix, and the eutectic network was also more evenly distributed within the matrix. The average grain size, measured using lineal intercept method following ASTM 112, is presented in Table 1 for all samples.

![SEM micrograph of cryorolled SAC305 (a) as-cast, (b) 0 dipping, (c) 10 min, (d) 30 min, (e) 60 min dipping, 1000x](image-url)

Fig. 2. SEM micrograph of cryorolled SAC305 (a) as-cast, (b) 0 dipping, (c) 10 min, (d) 30 min, (e) 60 min dipping, 1000x
From Table 1, SAC305 solder sample with dipping in liquid nitrogen for 60 minutes shows the largest grain size which is 12.50 μm. On the other hand, sample dipped for 30 minutes prior to cryorolled gave the smallest average grain size which is 5.88 μm. The 10 minutes dipping sample and sample without dipping exhibited average grain size value of 6.90 and 7.14 μm respectively. Meanwhile, average grain size of as cast sample is 11.00 μm which is 87% larger than the smallest grain size (5.88 μm) and this shows great potential of cryorolling for grain refinement of solder alloys. Longer dipping time of 60 minutes however, resulted in increment of grain size and it is also observed that the longer dipping time tend to increase brittleness of sample with small cracks started to appear at the edges during rolling. At this point of time, we are still unclear as to the exact reason why longer duration of dipping led to brittleness.

| SAC305 samples | Average grain size (µm) |
|----------------|-------------------------|
| As-cast        | 11.00                   |
| 0              | 7.14                    |
| 10             | 6.90                    |
| 30             | 5.88                    |
| 60             | 12.50                   |

Cryorolling was also observed to result in break-up of IMC particles resulting in smaller particles distributed more evenly throughout the β-Sn matrix. Figure 3 shows higher magnification of as-cast and 30 minutes dipping samples and it is clear that cryorolled sample had smaller Ag₃Sn particles compared to the as-cast sample. On the other hand, Cu₆Sn₅ particles might not have any changes in size as they are originally in small rounded shape in the as-cast solder. Smaller β-Sn grains were also more prominent in figure 3(b) compared to that of the as-cast sample. This shows another advantage of SPD, in that it is able to break up compound or particles resulting in more evenly distributed fine particles within the matrix. Finer particles, as has been commonly reported in literature, are more efficient in blocking movement of dislocation and thus, providing excellent improvement in mechanical properties.
3.2 Lap Joint Shear Strength

Solder samples were reflowed on bare pure copper substrate in the form of a single lap joint for shear strength test. The shear strength of all solder joints are given in Table 2. As can be seen, the strength increased from sample with 0 dipping to 30 minutes dipping before the strength decreased for the 60 minutes dipping sample.

| Dipping time | Ave. shear strength, MPa |
|--------------|--------------------------|
| 0            | 51.9                     |
| 10           | 50.6                     |
| 30           | 55.7                     |
| 60           | 48.8                     |

Cryogenic treatment has been reported to induce a large internal stress in the matrix of the material, which is the incentive of amounts of dislocations generation. This means that the strength and toughness of alloy will be improved because of the entanglement and pinning of these dislocations. With larger dislocation density, the mechanical properties of solder material will be enhanced [8, 10]. At the same time, cryogenic treatment suppresses dynamic recovery process during deformation, and thus effectively increase dislocation density without being erased by re-crystallization especially in low melting temperature alloy such as solder. Cross slip and dislocation annihilation processes are prevented because of the limited mobility of defect at such low temperature. Therefore, cryogenic treatment can be seen as a potential technique in increasing mechanical properties of solder.

The combination of cryogenic treatment and rolling also was observed to encourage grain refinement in the solder samples. Therefore, based on the Hall-Petch relation, this would help to improve the dislocation motion resistance, and thus, the increased dislocation density would result in improved strength in the solder. As grain size reduces, the yield strength will increase. From the microstructure observed under SEM and based on the average size for SAC305 solder samples in Table 1, sample with dipping in liquid nitrogen for 30 minutes has the smallest grain size. As listed in Table 2, this particular sample gave highest shear strength compared to sample rolled without prior dipping in nitrogen. In addition to that, as cryorolling also resulted in breaking up of large Ag3Sn particles, the solder could potentially has better toughness and possibly creep resistance. This comes from the more uniform distribution of small IMC particles which could act as pinning mechanism and improve the creep resistance. Work on creep behavior of cryorolled solder alloys is currently underway to evaluate how cryorolling influence the creep resistance of SAC305 alloy.

4.0 Conclusion

Several conclusions can be made in this work:

1. Cryorolling was observed to encourage grain refinement in SAC305, up to dipping for 30 minutes. Longer dipping time of 60 minutes resulted in larger grain size.
2. Cryorolling process also resulted in breaking up of particularly the Ag3Sn particles into smaller size. Smaller IMCs and more evenly distributed eutectic network was observed in cryorolled samples.

3. Shear strength of the solder joint improved in cryorolled samples due to smaller β-Sn grains, smaller IMC particles and more evenly distributed eutectic network. These would be more efficient in blocking dislocation movement leading to higher strength.

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