Development of Micro-structured Sn-2Ag-0.1Al Solder with Highly Thermal Fatigue Durability

Junichi Tanaka1, Toshiyuki Takashima2, Kenji Yoshimi3, Koji Serizawa3, Toshio Narita*

1 Graduate School of Engineering, Hokkaido University, Sapporo, Japan
2 Department of Mechanical Engineering, Hokkaido Institute of Technology, Sapporo, Japan
3 Hitachi, Ltd. Production Engineering Research Laboratory, Yokohama, Japan

*corresponding Author, narita@eng.hokudai.ac.jp

Abstract

Addition of small amount of Al suppressed super cooling of Sn-Ag solders completely under normal cooling rates and the Sn-2Ag-0.1Al solder has a fine structure with an average grain sizes less than 10 micron-meter. Stress-strain measurements showed that the Sn-2Ag-0.1Al solder have fracture strain and toughness superior than the conventional Sn-Ag solders and wetting of the Sn-2Ag-0.1Al solder was moderate. Thermal cycling test was carried for the Cu / ceramic module with a 200 micro meter Sn-2Ag-0.1Al solder sheet between 233 and 398K. Dor comparison purpose conventional solders as Sn-3Ag-0.5Cu were also tested under the same thermal cycling condition. It was found that the Sn-2Ag-0.1Al solder was still sound after the 300 T.C. in contrast to results for Sn-3Ag-0.5Cu failed completely after the 150 T.C.. The crack formed in the module with a Sn-2Ag-0.1Al solder propagated within the solder, but it was passed through the solder / Cu substrate interface for the module with a Sn-3Ag-0.5Cu. Micro-structuring process of the solder with Al addition could be discussed.

1 Introduction

Sn-Ag based alloys are candidate solders for various electric applications, replacing conventional Sn-Pb solders, because recent environmental consciousness raises a great concern on lead in solders used. The Sn-Ag and Sn-Ag-Cu solders consisted of a mixture of a primarily-coarse Sn crystal and rod-like precipitates of intermetallics Ag3Sn and Cu5Sn6, due to a super-cooling during solidification process, and these solders resulted in less fatigue durability during thermal cycling (T.C.), although they have relatively high mechanical strength at elevated temperatures. In our previous investigations addition of small amount of Al into Sn-based Pb-free solders changed its structure from a mixture of coarse Sn crystals and rod-like intermetallics precipitates to a fine precipitates and the primarily β-Sn phase with fine sub-grains [1]. It was found that the newly developed solder was superior in mechanical and fatigue properties.

In this investigation Sn-Ag-Al solders with 2.0 and 3.5Ag and different Al contents were prepared and their microstructures and mechanical properties were examined [2], and then by making use of these solders the joint sandwiched by Cu and ceramic plates with a power module package structure was prepared and thermal cycling test was carried out to evaluate durability of the newly developed Sn-Ag-Al solder by measuring crack initiation and propagation with thermal cycling between 233 and 398K for up to 500 cycle.

2 Experimental

Solders with or without Al addition was prepared in our laboratory by melting it in air, followed by solidified in a stainless steel mould with a cooling rate of 10K/s at solidifying temperatures from cooling curve measurements. Sn-(2.0 and 3.5) Ag solders were melted in air at 623K for 60min, while in a case of the Sn-2.0 Ag-(0.01 and 0.1) Al solders Al was added to Sn-Ag solder under stirring of the melt and kept for 60 min at 773K, and then the solders were cooled down to 623 K and after holding for 60 min it was solidified in the stainless steel mould.

Tensile tests was carried out at 298K with a strain rate of $10^{-4}$ using a specimen shown in Fig.1, which was machined

![Fig.1 Schematic of the specimen for tensile test.](image1)

![Fig.2 Schematics of the specimen after soldering.](image2)
from the ingot (30mm diameter and 200 mm length), and a
gauge length (around 50 mm) was monitored by using a
CCD camera and computer system. Microstructures of
fracture, polished, and cross-cut sections were observed
using SE. Etching in a 3% hydrochloric acid alcohol solution
for 5 to 60 sec. was adopted to reveal microstructures of the
solders. The hardness was measured using a Shimadzu
Micro-Vickers hardness tester under a 100g load for xx sec.
The solders with 200µm thick was used to prepare the Cu /
Ceramics joint, where Ni with about 3 micron meter was
electro-plated on the Cu surface, and a low activated rosin
flux was used, and the joint was heat cycled, as shown in
Fig.2. Crack length was measured by cutting the joint along
its diagonal line.

3 Results and discussion

Fig.3 shows a microstructure of the as-solidified
Sn-2%Ag-0.1%Al solder and an enlarged structure of the
grain in Fig.3(a) is given in Fig.3(b). It was found in Fig.3
that a sub grain structure formed in the primarily β-Sn
Crystal. Furthermore, an area fraction the eutectic phase
decreased in comparison to the Sn-2%Ag solder as shown
in Fig.3(c) and there are two intermetallics of Ag3Sn and
Ag2Al in the Sn-Ag-Al solder.

Stress-strain curves in Fig.4 could be divided into three
regions, an initial increase in strain up to ultimate strength as
1st region, a final rapid decrease in stress as a 3rd region and
2nd region between these two regions. As shown in Fig.5,
the Sn-Ag-Al solder showed long time for the 2nd region, and
this seems to be due to a fact that Crystal size of Sn became

Fig.3 Microstructure of (a) Sn-2%Ag-0.1%Al and (c)Sn-2%Ag.

Fig. 4 Stress-Strain curves of each alloys.

Fig. 5 Strain that is divided for each inflection point.

Fig. 6 Microstructure of fractured surface.
(a) Sn-3%Ag-0.5%Cu (b) Sn-2%Ag-0.1%Al
finer by adding Al, forming sub-grain structure. Al addition of 0.1wt% was enough, but 0.05wt%Al id show little effect.

Figure 6 shows microstructure of the fractured surface observed by SEM and morphologies could be summarized as follows. Trans-granular cracks through a primarily β-Sn crystal formed in parts of the Sn-3%Ag-0.5%Cu eutectic alloy. Dendrite main-stems were clearly seen as well. Dimples formed on Sn-2%Ag-0.1%Al hypo-eutectic alloys, suggesting that the fracturing process proceeded irrespective of inter and intra crystals.

Figure 7 shows a polished microstructure cut parallel to a tensile stress loading direction. In case of the Sn-3%Ag0.5%Cu eutectic alloy the primary β-Sn crystal was found to be stretched significantly. While, both a primarily β-Sn and an eutectic structure stretched evenly.

Figure 8 shows change in crack length measured for the Cu / ceramic modules during the heat cycling. It was found that the length of crack against number of cycles.

(a) Sn-3%Ag-0.5%Cu (b) Sn-2%Ag-0.1%Al

Fig. 7  Microstructure cut parallel to loading direction.  
(a) Sn-3%Ag-0.5%Cu (b) Sn-2%Ag-0.1%Al

Fig. 8  Total Length of Crack (mm) against Number of Cycles.

(a) Sn-2Ag-0.1Al  (b) Sn-3Ag-0.5Cu

Fig. 9  Morphology of the crack propagation.

(a) Sn-2Ag-0.1Al  (b) Sn-3Ag-0.5Cu
that crack of the Sn-Ag-Al solders propagated slower than the Sn-3Ag-0.5Cu, showing an superior thermal fatigue property. The crack propagation rates of Sn, Sn-0.1Al and Sn-0.2Al solders are between those of the Sn-3Ag-0.5Cu and the Sn-Ag-Al solders.

As shown in Figure 9, the crack of the Sn-3Ag-0.5Cu propagated along the interface between the Cu plate and the solder, while the Sn-2Ag-0.1Al solder cracked inside of the solder. In case of the Sn-0.1Al solder cracks propagated along the ceramic / solder interface.

4 Conclusion

Addition of Al into Sn-2Ag solder formed a fine structure of a primarily Sn grain with sub-grains and the sub-grain size was 5 micron meter, which was one fifth of the conventional Sn-3Ag-0.5Cu solder. Measurements of stress strain curves showed that fracture strain of the solder containing Al increased in contrast to those of the Sn-3Ag-0.5Cu without decrease in ultimate tensile strength. Fracture strains tended to increase with decreasing Al content from 0.1 to 0.05mass% irrespective of Ag contents. Thermal cycling tests were carried out for the Cu/ceramic module at temperatures between 233 and 398K up to 300 cycles. It was found that crack propagation rates decreased for Sn-3Ag-0.5Cu, pure Sn and Sn-Ag-Al solders in this sequence.

5 Literature