Reducing Cracking in Solder Joint Interfacial Cu₆Sn₅ with Modified Reflow Profile

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Abstract

The polymorphic transformation that occurs in the Cu₆Sn₅ intermetallic compound (IMC) at 186°C has the potential to generate stresses that could lead to cracking of that phase in soldered joints during the multiple reflow cycles of a typical printed board assembly process and the thermal cycles to which electronic assemblies are exposed during service. In this paper the authors report on the effect of variations in the cooling stage of a reflow soldering thermal profile on the incidence and extent of cracking in the Cu₆Sn₅ at the interface between solder alloys and copper substrates. The solder alloy/substrate combinations studied were Sn-3.0Ag-0.5Cu/Cu and Sn-0.7Cu-0.05Ni-1.5Bi/Cu. The cooling conditions were (i) the direct-cooling of a conventional reflow profile, and (ii) an alternative reflow profile with one of three extended isothermal holding periods of 30, 60, and 180 seconds at 140°C during the cooling stage. It was found that the alternative reflow profiles reduced cracking in the interfacial Cu₆Sn₅ IMC layer and this resulted in improved resistance of the reflowed solder ball to failure in high speed impact shear when the distribution of stress tends to favor crack propagation though the interfacial IMC rather than through the bulk solder.

Keywords: Sn-3.0Ag-0.5Cu, Sn-0.7Cu-0.05Ni-1.5Bi, Time-Temperature-Transformation, Microstructure, Reliability

1. Introduction

Miniaturisation of electronic circuitry and the consequent trend toward finer pitch interconnects has meant that the reliability of the final assembly is increasingly dependent on the integrity of very small solder joints. The increased use of multiple reflow cycles in the assembly of complex circuitry means that it is important that the joints retain their integrity through repeated reflow profiles.[1–5]

In the absence of other factors the IMC that forms at the interface between Sn-based lead-free solder and a copper (Cu) substrate in the reflow process is Cu₆Sn₅. Above 186°C the stable form of Cu₆Sn₅ is the hexagonal η phase.[6] At temperatures below 186°C the stable form of Cu₆Sn₅ is the monoclinic η’S phase.[7] It has been hypothesized that when the Cu₆Sn₅ is constrained within a solder matrix the approximately 2.15% volume change that is associated with the η ↔ η’S transformation may result in the buildup of stress that could lead to cracking of Cu₆Sn₅, particularly when the joint is subjected to multiple reflow cycles.[8, 9] However, the magnitude of the stresses generated by the polymorphic transformation and the effect on the reliability of the solder joint are not yet fully understood.

Cooling rates during reflow profiles are not usually considered to have a significant effect on joint reliability with 60–240°C/min being the typical range.[10] For a non-eutectic lead-free solder such as the Sn-3.0Ag-0.5Cu alloy that has a freezing range, faster cooling rates of 180-240°C/min are recommended to achieve a finer grain structure and reduce shrinkage cracking. In setting these cooling rates, no consideration has been given to the possible consequences of the polymorphic transformation of the Cu₆Sn₅ that is always present at the solder/Cu inter-
face. Using a time-temperature transformation (TTT) diagram recently developed through the use of in-situ high-voltage transmission electron microscopy (HV-TEM),[11] the effect of cooling conditions was systematically studied with a view to confirming whether the cooling profile could have an effect on the integrity of the interfacial Cu₆Sn₅.

The basis of the experimental approach taken in this work is the observation that the volumetric change associated with the $\eta \leftrightarrow \eta'$ transformation varies with the temperature at which the transformation occurs. On the basis of that observation, it is hypothesized that if the transformation occurs at a temperature at which the difference in the volume of the two phases is close to zero, there should not be sufficient stress generated to initiate cracking. This hypothesis is illustrated schematically in Fig. 1 (a), which is based on previous dilatometry measurements.[12] The TTT diagram for the Cu₆Sn₅ transformation derived from these studies is shown in Fig. 1 (b). In the experiments reported in this paper this hypothesis is tested by reflowing solder balls to a copper substrate with the two different cooling conditions shown schematically in Fig. 1 (c). One is a conventional reflow profile and the other an alternative profile with an extended isothermal holding stage. For the conventional reflow profile (direct-cooling), the cooling rate chosen was around 30°C/min (Fig. 1 (b)). This rate is at the low end of the range usually used in commercial reflow but it ensures that the test piece reaches 140°C before the TTT diagram predicts that the polymorphic transformation will have begun. In the experiments reported in this paper the focus was on the effect of isothermal interruptions of a cooling rate that takes the temperature-time plot close to the transformation ‘nose’ of the TTT diagram, which indicates the temperature at which the transformation has the earliest onset. The expectation is that when cooling at this rate from a temperature of around 240°C to room temperature (RT), the extent of the $\eta \rightarrow \eta'$ transformation will be affected by the inclusion of the isothermal hold. The plot in Fig. 1 (a) indicates that when the transformation occurs in the temperature range between 140–160°C there will be minimal change in the unit cell volume of Cu₆Sn₅ as it transforms from the $\eta$ to the $\eta'$ form.[13] The objective is to confirm that the effect occurs and to determine the time required for the completion of transformation at that temperature.

In this paper, we focus on the effect of various reflow cooling conditions on the reflow soldering of a solder ball to a Cu substrate using two lead-free solder alloys (in wt%), Sn-3.0Ag-0.5Cu and Sn-0.7Cu-0.05Ni-1.5Bi. Sn-0.7Cu-0.05Ni-1.5Bi was chosen as the comparison alloy because the Ni in this alloy incorporates into the interfacial IMC and stabilized the $\eta$ form of this phase, which is characterized as (Cu,Ni)₆Sn₅.[9] These two solder alloys are reported to have similar mechanical properties[14] so that any significant difference in the response of the reflowed test piece to shear impact should be attributable to the effect of the isothermal hold on the integrity of the IMC layers. The Sn-0.7Cu-0.05Ni-1.5Bi alloy has recently been the subject of intensive testing because of the stability of the solid solution strengthening during exposure to elevated temperatures.[3, 4, 14–18] The Sn-3.0Ag-0.5Cu alloy

![Fig. 1](image-url)
has become the default Pb-free solder, particularly for reflow soldering. These two solders thus provide an ideal basis for assessing the effectiveness of controlled cooling after reflow in improving the reliability of lead-free solder joints.

2. Experimental

Approximately 500 μm diameter balls of each alloy were reflowed onto 500 μm diameter Cu pads that had an organic solderability preservative (OSP) surface finish. Reflow was carried out in a benchtop reflow oven simulator (BT 300 series, Mannecorp) with the aid of a small amount of no-clean flux. The oven used in this study was a single-zone reflow with multiple-stage temperature control system. The oven uses both infrared and hot-air convection systems as the heating sources. The temperature accuracy of the reflow oven was ±2°C. The time above 220°C was fixed at ~110 s for all reflow profiles. In the reference reflow profile the reflowed solder ball cooled from 240°C to room temperature at a rate of about 30°C/min. The temperature curves shown in Fig. 2 are plots of the actual temperature experienced inside the oven during the reflow cycles. For the cooling system, two axial flow fans force cooled air in the oven. The temperature gradient during cooling was controlled by setting the cooling step in stages to maintain the desired cooling rate at ~30°C/min as it reached the desired isothermal condition of 140°C. To determine the effects of multiple reflows some samples were reflowed 2 and 8 times.

Reflowed samples were then cross-sectioned and fine polished perpendicular to the solder/Cu interface. The solder joints were observed using a desktop scanning electron microscope (SEM) (TM3030, Hitachi, Japan). The average thickness of the total IMC layer, \( d_{ave} \), was determined by the following equation:

\[
d_{ave} = \frac{A}{L}
\]

where \( A \) is the total area of the IMCs as shown schematically with hatching in Fig. 3, and \( L \) is the IMC length horizontally across the interface. The total area of cracking was normalized to the total measured \( L \). The quantitative image analysis was done using ImageJ software (NIH, USA).[19]

Ball shear tests were performed at shear speeds of 10 mm/s and 2,000 mm/s using a DAGE-4000s bond microtester (Nordson, USA). At least 20 reflowed solder balls were tested for each test condition. The hammer height above the substrate was fixed at 50 μm. After the shear test, fracture surfaces were observed using SEM and classified according to the fracture mode.
3. Results and Discussion

3.1 Influence of reflow cooling conditions on IMC microstructure, growth and cracking at the solder/Cu interface

Figure 4 shows representative microstructures after eight reflow cycles with direct-cooling (0 s holding at 140°C) and interrupted-cooling (30 s, 60 s, and 180 s holding at 140°C). It is well-known that in the Sn-3.0Ag-0.5Cu alloy the IMC that forms at the interface is Cu$_6$Sn$_5$[3–5] while in the Sn-0.7Cu-0.05Ni-1.5Bi alloy the IMC at the interface is (Cu,Ni)$_6$Sn$_5$.[9, 15, 18] The Cu$_6$Sn$_5$ has a larger grain size than the (Cu,Ni)$_6$Sn$_5$. The microstructures in Fig. 4 (b) are representative of the bulk solder in the Sn-3.0Ag-0.5Cu after eight reflows with the four cooling conditions. The eutectic region in the Sn-3.0Ag-0.5Cu/Cu bulk microstructure consists of finely dispersed Ag$_3$Sn in a Sn matrix.[4] In Sn-0.7Cu-0.05Ni-1.5Bi/Cu solder joints, the primary (Cu,Ni)$_6$Sn$_5$ IMCs coarsens when reflowed with interrupted-cooling at 180 s.

Figure 5 compares the average interfacial IMC thickness over multiple reflow cycles. In all cooling conditions the interfacial IMC thickness increases with the number of reflow cycles. It can be seen that (Cu,Ni)$_6$Sn$_5$ grows more slowly than the Cu$_6$Sn$_5$. Evidence suggests that a thicker and coarser IMC layer is associated with earlier failure in thermal cycling.[20–22] In direct-cooling the average thickness of the interfacial Cu$_6$Sn$_5$ layer at the Sn-3.0Ag-0.5Cu/Cu interface grows from ~3.6 μm after the first reflow cycle to ~7.6 μm after eight reflow cycles, while in the Sn-0.7Cu-0.05Ni-1.5Bi/Cu the (Cu,Ni)$_6$Sn$_5$ layer grows from ~3.4 μm to a maximum ~6.9 μm after eight reflow cycles. In test pieces reflowed with interrupted-cooling the growth of the interfacial layer increases with longer holding times (>30 s) at 140°C for both alloys. In the Sn-0.7Cu-0.05Ni-1.5Bi reflowed with holding times of 30 s and 60 s there is much less IMC growth after eight reflow cycles.
than with direct-cooling. The (Cu,Ni)6Sn5 layer grows to a maximum thickness of ~4.3 μm after eight reflow cycles with interrupted-cooling for 30 s, which is ~37% thinner than the IMC in the direct-cooled samples. This suppression of IMC growth in multiple reflow with interrupted-cooling did not occur in Sn-3.0Ag-0.5Cu/Cu. Significantly, after eight reflow cycles the average IMC thickness growth in Sn-3.0Ag-0.5Cu/Cu reflowed with cooling interrupted for 30 s and 60 s is no greater than the average IMC thickness in direct-cooled samples after eight reflow cycles.

Figure 6 shows the normalized crack area in IMCs for both alloys. Significantly fewer cracks occur in the interfacial (Cu,Ni)6Sn5 IMC at the Sn-0.7Cu-0.05Ni-1.5Bi/Cu interface than in the Cu6Sn5 at the Sn-3.0Ag-0.5Cu/Cu interface (Fig. 4). For both alloys, no cracks were observed in the interfacial IMC if the solder balls were reflowed with cooling interrupted for 30 s. Ni is known to stabilize the hexagonal form of Cu6Sn5 so that Ni-containing solders generally show less cracking in the interfacial IMCs.[8, 9] Increasing the isothermal holding time beyond 30 s results in thickening and cracking of the interfacial IMC. This could be due to stress induced by the increasing growth of the η’ phase at 140°C. It has recently been shown by Wieser et al.[23] that there is a spontaneous strain in η’-Cu6Sn5 phase due to the monoclinic lattice distortion and they suggested it could significantly influence the reliability of solder joints. Longer holding times at 140°C also may have promoted stress induced by the η’→η transformation during heating in the second cycle and subsequent cycles.

These results provide clear evidence of the effectiveness of controlled cooling as a means of minimizing cracking in Cu6Sn5 at the interface between the Cu substrate and solder alloys that do not contain Ni.

In summary:
For Sn-3.0Ag-0.5Cu reflowed to a Cu substrate;
(i) The crack area in the interfacial IMCs increases with multiple conventional reflow cycles.
(ii) No cracks were observed in the IMCs in samples reflowed with a thermal profile that includes a 30 s interruption at 140°C.
There was a small reduction of the crack area in the 1st reflow cycle with interrupted-cooling for 60 s and 180 s compared to direct-cooling, but the cracking worsens with further reflow cycles.

For Sn-0.7Cu-0.05Ni-1.5Bi/Cu:
(i) The area of cracking is much less in all condition compared to Sn-3.0Ag-0.5Cu/Cu.
(ii) No cracks were observed with interrupted-cooling for 30 s.
(iii) Multiple reflows with cooling interrupted for >30 s do not increase the extent of cracking.

3.2 Shear-impact performance

At shear speeds ≥ 1,000 mm/s the crack propagates partially or completely through the solder/substrate interface so that the result provides a measure of the strength of the interfacial intermetallic.[24–26] At shear speeds of ≤ 100 mm/s when the crack propagates through the ball the result provides a measure of the properties of the bulk solder. In this study, the joint strength of both solder alloys was evaluated using shear speeds of 2,000 mm/s and 10 mm/s. The shear test results for both Sn-3.0Ag-0.5Cu/Cu and Sn-0.7Cu-0.05Ni-1.5Bi/Cu after the first, second and eighth reflow cycles are summarized in Fig. 7. The grey bars represent the average values for the maximum force required for fracture or detachment of the solder ball. This force is indicative of the strength of the bond between the refloved solder ball and the Cu substrate. The red lines show the average amount of energy absorbed during the shearing of the solder joint.

The results of shear impact testing at a speed of 2,000 mm/s indicate that the variation in reflow cooling conditions does not have any significant effect on the maximum force in Sn-0.7Cu-0.05Ni-1.5Bi/Cu but it has to be acknowledged that the large error bars diminish the significance of these results. In contrast, for the Sn-3.0Ag-0.5Cu/Cu samples there is a noticeable tendency for the shear strength to be improved by the interrupted-cooling after multiple reflows. For example, the shear strength in Sn-3.0Ag-0.5Cu/Cu reflowed with direct-cooling is 9.52 ± 0.89 N after eight reflow cycles but improves to a maximum of 14.15 ± 0.94 N when reflowed eight times with interrupted-cooling for 180 s. Although the best IMC formation with low thickness and no cracking was observed in Sn-3.0Ag-0.5Cu/Cu with interrupted-cooling for 30 s samples, the shear strength is slightly lower at 12.32 ± 0.90 N after eight reflow cycles.

In this study, the samples tested in high speed shear test were classified into four fracture modes, i.e. brittle, quasi-brittle, quasi-ductile, and ductile (Fig. 8 (a)). The result shows that after eight reflow cycles, the quasi-brittle failure mode was more dominant in Sn-3.0Ag-0.5Cu/Cu while ductile fracture was the predominant mode in Sn-0.7Cu-0.05Ni-1.5Bi/Cu. Multiple reflows resulted in a gradual decline in the amount of absorbed energy during shear.
testing at high speed shear tests for both alloys. In all conditions more energy is required to fracture the Sn-0.7Cu-0.05Ni-1.5Bi/Cu than Sn-3.0Ag-0.5Cu/Cu (Fig. 7). This is evidenced in the failure mode analysis in Fig. 8 (b). The Sn-0.7Cu-0.05Ni-1.5Bi/Cu samples show a greater tendency to quasi-ductile or ductile fracture than Sn-3.0Ag-0.5Cu/Cu. Ductile fracture is usually associated with greater energy absorption.

Figure 9 shows the variation of maximum force as a function of the interfacial IMC thickness and reflow cool-
ing conditions in (a) Sn-3A-0.5Cu/Cu and (c) Sn-0.7Cu-
0.05Ni-1.5Bi/Cu. The general trend is that the maximum
force decreased with increasing interfacial IMC layer
thickness for both solder alloys and all cooling conditions.
Since the IMC thickness has a strong influence on the sol-
der ball shear strength at 2,000 mm/s, the Sn-0.7Cu-
0.05Ni-1.5Bi/Cu solder joints performed better at high
speed shear test because of their lower interfacial IMC
layer thickness. Figure 9 (b, d) shows typical fracture sur-
faces of both solders after eight reflow cycles with inter-
rupted-cooling for 180 s. In this condition, average IMC
thickness is the largest for both alloys (Fig. 5 (a, b)). From
the fractographs and failure mode analysis in Fig. 8, it is
clear that the Sn-3.0Ag-0.5Cu/Cu joints have a tendency to
fail in a quasi-brittle mode. This is apparent in the exposed
IMC layer with some remnant of the solder ball left on the
fracture surface. Quasi-brittle mode involves fracture in
>50% area of the brittle IMC layer area and a smaller area
of ductile solder. This means that the bulk solder just
above the IMC layer may be playing a role in the fracture
process. Hence, further studies should be done including
an investigation of other factors that might affect the maxi-
mum shear force in relation to the shear test results.
It can also be seen in Fig. 9 (d) that the fractured sur-
facer in quasi-brittle Sn-0.7Cu-0.05Ni-1.5Bi/Cu has more
ductile Sn-rich features in the remnants of the solder ball
due to finer (Cu,Ni)₃Sn₅. As shown in Fig. 8 (b), more than
50% of the Sn-0.7Cu-0.05Ni-1.5Bi/Cu test pieces failed in
quasi-ductile to ductile fracture modes. This could explain
the higher absorbed energy in Sn-0.7Cu-0.05Ni-1.5Bi/Cu
compared to Sn-3.0Ag-0.5Cu/Cu. Consistent with the
hypothesis on which this experimental program was based
there is a clear tendency for Sn-3.0Ag-0.5Cu/Cu to per-
form better in high speed shear tests when the reflow
cycle is interrupted with an isothermal holding stage even
though a thinner interfacial IMC layer thickness was
observed in direct-cooled samples. This effect is more
apparent in samples reflowed with interrupted-cooling for
180 s. This is presumably because, even though the IMC
layer is thicker, it has less cracking because the \( \eta \rightarrow \eta' \)
transformation occurs at a temperature at which the vol-
ume change is at a minimum.
Apart from the \( \eta \rightarrow \eta' \) transformation and IMC growth,
the Ag₃Sn particles in the bulk solder microstructure are
also susceptible to coarsen over multiple reflow cycles
with longer holding times at 140°C. It has been frequently
reported that the Ag₃Sn coarsening tends to weaken the
mechanical properties of solder joints.[3, 4, 27–29] To
assess the bulk solder properties, a low shear impact
speed test of 10 mm/s was done on both solder joint sam-
pies. As shown in Fig. 7 (c, d), multiple reflow cycles with
different cooling conditions do not significantly affect the
results for either alloy. The maximum force reached in the
shearing of the Sn-3.0Ag-0.5Cu/Cu and Sn-0.7Cu-0.05Ni-
1.5Bi/Cu samples is similar despite the variation in reflow
cooling conditions. From the SEM micrographs shown in
Fig. 10 and shear result in Fig. 7 (c), it can be seen that the
longest holding time at 180 s with interupted-cooling con-
dition used in this study may not adequately coarsen the
Ag₃Sn compounds to the point that it compromises the
bulk solder strength.
This study has shown the benefit of considering the
TTT-curve of the \( \eta \leftrightarrow \eta + \eta' \)-Cu₆Sn₅ transformation in
designing a reflow soldering profile. It is proposed that in
alloys that do not have the benefit of a Cu₆Sn₅ phase that
has been stabilized by Ni, the cooling can be managed to
provide an opportunity for the transformation to occur at a
temperature at which the change in unit cell volume is at a
minimum. By using this interrupted-cooling method in the
reflow process, it is possible to prevent early cracking in
the interfacial IMC layer during the assembly process.

4. Conclusion

The effect of various cooling conditions on the microstructure and shear impact properties of Sn-3.0Ag-0.5Cu/Cu and Sn-0.7Cu-0.05Ni-1.5Bi/Cu solder joints has been studied. To minimize cracking in the solder joint, a new alternative reflow cooling condition is proposed to replace the conventional direct-cooling. It is found that cracking in the interfacial Cu₆Sn₅ can be inhibited with an isothermal holding step of 30 s at ~140°C during cooling from 240°C at a cooling rate of 30°C/min. Based on IMC thickness results after eight reflow cycles, not only does the 30 s hold not further thicken the IMC layer in Sn-3.0Ag-0.5Cu/Cu solder joints, but it also significantly reduces the thickness of the IMC layer in Sn-0.7Cu-0.05Ni-1.5Bi/Cu joints. As could be expected on the basis of the stabilizing effect of the Ni, Sn-0.7Cu-0.05Ni-1.5Bi/Cu consistently showed better shear strength than Sn-3.0Ag-0.5Cu/Cu, and the variation in cooling conditions does not affect its performance in ball shear testing at 2,000 mm/s and 10 mm/s. Shear strength performance in Sn-3.0Ag-0.5Cu/Cu solder joint can be significantly improved with the proposed alternative reflow profile.

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