Mechanical Reliability of Aged Lead-Free Solders

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2012

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<td>Project title: Reliability of Aged Lead-Free Solders</td>
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Abstract

The usage of lead-free solder joints in electronic packaging is of greatest concern to the electronic industry due to the health and environmental hazards arising with the use of lead. As a consequence, lead is legally prohibited in the European Union and the industry is aiming to produce lead-free products.

The reliability of solder joints is an important issue as the failure could destroy the whole function of a product. SnAgCu is a commonly used alloy for lead-free solders. Compared to solders containing lead, tin-rich solders react more rapidly with the copper substrate. The reaction results in formation of brittle intermetallic compounds and in poor mechanical reliability. The formation can be slowed down by the addition of nickel in the under bump metallization.

In this project the objective was to evaluate the mechanical reliability of solder joints in high temperature applications. An alloy of nickel and phosphorus was plated on copper plates by electroless plating. The plates were joined together using SnAgCu solder. The samples were then thermally aged at 180 °C for different durations (100, 200, 300, 400 and 500 hours). Tensile tests were performed on the samples. The result from the tensile test showed a decrease in mechanical strength with increasing aging duration. The fracture path shifted from being in the bulk solder to being at the interfaced.

Keywords:
Solder joint, SnAgCu, lead-free, electroless NiP, tensile test, thermal aging
Acknowledgement

The author of the following report would like to acknowledge the guidance, support and assistance during this project.

First I would like to thank my supervisor Professor Chen Zhong at the School of Material Science and Engineering at Nanyang Technological University (NTU) for his guidance and support throughout this project. I would also like to thank PhD student Ms Yang Ying for her help and assistance during the experiments and the technical staff of Ms Yeow Swee Kuan and Mr Patrick Lee for assistance and advices during laboratory experiments.

Furthermore, I would like to take the opportunity to thank Professor Anders Eliasson, Department of Material Science and Engineering at the Royal Institute of Technology (KTH) for guidance and support. His comments and guidance have been very helpful and appreciated. I would also like to thank him for handling the logistics during the project.
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## Nomenclature

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<tr>
<td>UBM</td>
<td>Under bump metallization</td>
</tr>
<tr>
<td>SnAgCu solder</td>
<td>Solder containing an alloy of 95.5 wt% tin, 4 wt% silver and 0.5 wt% copper.</td>
</tr>
<tr>
<td>SnAg solder</td>
<td>Solder containing an alloy of 96.5 wt% tin and 3.5 wt% silver.</td>
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<tr>
<td>NiP UBM</td>
<td>An alloy of nickel and phosphorus, used between the substrate and the solder to slow down the interfacial reaction.</td>
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<tr>
<td>Electroless NiP</td>
<td>A chemical process used to deposit a layer of NiP on the surface of a metal</td>
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<td>UTS</td>
<td>Ultimate tensile strength</td>
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1. Introduction

1.1 Background

Electronic packaging provides electrical and mechanical connection in the electronic system. It also protects the chip from the environment, for example from moist and dusts [1].

The solder joints of the components in electronic systems are usually the cause of failure in devices used for high temperature applications [2][3]. This makes the reliability of solder joints an important issue to the electronic industry. The most commonly used techniques to join the chip to its substrate are wire bonding and solder bumping. Because of the development towards smaller size and higher functionality of electronic devices, such as mobile phones and digital cameras, the demand is increasing for more input/output (I/O) connections. Wire bonding cannot meet the requirements but solder bumping technology make it possible to meet the demands. In solder bumping technology, it is possible to place a large number of solder bumps on the surface. However, since the number of solder bump connections increases, the size must decrease for the I/O connections to fit on the surface. This makes the reliability of the solder bumps even more critical for the product to function [1].

The structure of a solder joint can be seen in figure 1.1. The under bump metallization (UBM) is found between the solder and the metallic substrate. UBM consists of one or more layers of thin films on the I/O substrate. It provides a good wetting surface to the solder and slows down the reaction between the chip metallization and the solder [4].

![Figure 1.1 Structure of solder joint](image)

The Eutectic alloy of tin and lead has long been commercially used as material in solders. Lead has been used because of its suitable properties such as high density, high deformability and low melting point. It provides the tin-lead solder with a lower melting temperature, suitable for soldering. However, lead is a well-known toxic metal [6]. Electronic devices can end up in the waste site and the lead will pollute the environment. As a consequence, the use of lead in electronic devices has been prohibited in the European Union due to the Restrictions of Hazardous Substances Directive (RoHS) that took affect 2006 [7][8]. The electronic industries now aim to produce lead-free products and research on suitable alternative alloys is ongoing.
One of the problem arising when lead is removed from the solders is that during reflow the under bump metallization layers react with the solder and form intermetallic compounds. They are normally brittle, which results in poor mechanical strength and the risk of failure increases. The mechanical reliability of the solder joints is an important aspect in high temperature electronic packaging (150-200°C). To slow down the formation of intermetallic compounds and to improve the wetting, different alloys are studied as both for solders and UBM [4].

SnAgCu is a ternary alloy with desirable properties and commonly used for lead-free solders. However, the tin-rich solders react more rapidly with the copper UBM compared to lead-containing solders. The reactions results in formation of brittle intermetallic compounds and in poor mechanical reliability. The formation can be slowed down by the addition of nickel in the UBM because the reaction between nickel and tin is slower than between copper and tin [9] [10]. Electroless nickel-based UBM have been used for this purpose because of its suitable properties, good wettability and low cost [4].

Another commonly used alloy for lead-free solders is the binary eutectic alloy Sn-3.5Ag. This alloy and its mechanical properties have been widely studied combined with nickel and phosphorus UBM. The research has been made using tensile test to study the mechanical reliability [4] [9] [12] [11]. In this report the solder is SnAgCu but UBM of nickel and phosphorus will be used. The report concentrates on the mechanical properties after thermal aging. The solder in the samples will be an alloy containing 95.5wt% Sn, 4wt% Ag and 0.5wt% Cu. The substrate is made of copper and plated with electroless NiP. In order to evaluate the mechanical reliability of the NiP UBM a tensile test will be conducted on the samples after thermal aging.
1.2 Objective
The objective in this project is to study the mechanical reliability of lead-free solders in high temperature applications. The solder of the samples is made of an alloy with Sn (95.5wt%), Ag (4 wt%) and Cu (0.5wt%). The substrate is made of copper and plated with electroless NiP. The samples will be thermally aged on a fixed temperature (180 °C) for long durations (100, 200, 300, 400 and 500 hours). After aging, a tensile test will be conducted on the samples. The tensile test will show the mechanical strength and the fracture behavior of the samples. Thus it will be possible to analyze how high temperatures affect the mechanical properties of Cu/NiP/SnAgCu solder joints.

The method has previously been used on Cu/NiP/SnAg solder joints and the results from this report will be compared to those results.
2. Background
In this project the mechanical properties of Cu/NiP/SnAgCu solder joints will be examined. This part of the report covers background information from literature and previous research on electroless plating, intermetallic compounds and mechanical properties of solder joints. The experimental method used in this project has previously been used on NiP/Sn3.5Ag solder joints. This research will also be covered in the literature review.

2.1 NiP & Electroless Plating
There are several techniques being used to deposit nickel-based UBM on metallic substrate such as electroless plating, sputtering and electrolytic plating. The most commonly used is electroless plating due to its low cost and ease of control. Electroless NiP is an alloy of nickel and phosphorus and has good corrosion, wear and abrasion resistance. The microstructure of the alloy changes from microcrystalline to amorphous with increasing phosphorus content. Hence, the properties of the plated alloy depend on the phosphorus content, which normally is in the range of 6-13 wt% [13].

Electroless plating is a chemical reduction process used to deposit a layer of NiP on the surface of a metal. The plating solution includes one reducing agent such as hypophosphite. The hypophosphite reacts with the metal ion to deposit metal by a catalytic reduction [9] [4] [13].

2.2 Intermetallic Compounds
The mechanical reliability of solder joints is affected by the intermetallic compounds formed during the reactions in the solder/substrate interface. The forming of intermetallic compounds is inevitable during soldering, and it ensures a good metallurgical bond to the substrate. However, they are often brittle and cause a region of weakness that can result in failure. The formation can be enhanced by a long reflow time and high reflow temperature, but also by thermal aging or long-time storage. It can also be affected by long-term usage of the product at room temperature. The thickness increases with the reaction time, and the intermetallic compounds continue to grow during solid-state aging [4][14].

Consequently, thermal aging is a suitable method for testing the mechanical reliability of solder joints. During the process of thermal aging intermetallic compounds grow continuously due to element diffusion. Having a suitable under bump metallization is important for the development of reliable packaging technology since it slows down the reaction between the solder and the substrate. Copper based UBM works well with lead-tin solders. But lead-free solders, with a high content of tin, have a more rapid consumption of copper that result in formation of brittle intermetallic compounds. Therefore, nickel-based UBM are examined to slow down the reaction [4][9][14][15].
The driving force for the element diffusion is the nickel concentration gradient between the electroless NiP layer and the solder. The concentration of nickel is higher in the NiP layer and this makes the nickel atoms diffuse from the NiP layer towards the solder. Tin atoms diffuse from the solder towards the metallization. When the diffusing elements encounter, the elements react and form intermetallic compounds. Shohji et al. [12] made impact tests on SnAgCu solder joints after aging at 150°C up to 1000 hours. The results shows that mainly Cu₃Sn and Cu₅Sn₅ are the intermetallic compounds that are impacting the mechanical strength. [16] [10]. In figure 2.1 the formation between Sn3.5Ag0.5Cu solder and the Cu substrate is shown.

Figure 2.1 Formations of intermetallic compounds in the solder joint interfaces of Sn3.5Ag0.5Cu solder joint. [10]
2.3 Mechanical Properties
Solder joints work as interconnects in electronic packages and provide electrical and mechanical stability. In the evaluation of the reliability of solder joints a number of factors should be considered such as how the stress is distributed and the strain rate. Also, other environmental factors should be considered such as temperature. Some possible reasons for mechanical solder failure in use could be creep, fatigue, corrosion, formation of intermetallic compounds, voids, and electron migration [14] [1].

To examine the mechanical behavior and to characterize the failure of solder joints, several methods could be used such as; fatigue test, tensile and shear test, and three/four point bending test. Tensile and shear test investigate the load bearing capacity of solder joints [6]. In previous research, mainly shear test has been used and it has been routinely performed of the electronic industry [16]. In this project tensile test is used because it preserves the fractured surfaces, which can be used for fracture analysis. Also, the distribution in all interface layers is the same so the test can be used to reveal the weakest layer or interface [4]. The method used in this project for tensile testing has previously been used by the research group of Chen et al. [4].
2.4 Fracture Behavior of Sn3.5Ag/NiP Solder Joints After Tensile Test

The method of tensile testing for solder joints has been used in this report to evaluate the mechanical properties of solder joints and has previously been used by Chen et al. [4][9] at Cu/Sn3.5Ag/NiP solder joints.

The four different types of failures are:

a) Ductile fracture inside the bulk solder.
b) Dimpled interface failure between the solder and the intermetallic compounds.
c) Failure through interfacial layers.
d) Failure between the NiP coating and the substrate.

![Figure 2.2. Examples of the different failures, represented by a)-d) [1]](image)

The general trend is with extended aging duration and an increase of aging temperature, the fracture shifts from inside the bulk solder to the interface, shown in figure 2.2.

The Mechanical strength decreased in research by Chen et al. [9] on Cu/NiP/Sn3.5Ag solder joints, and is shown in figure 2.3.

![Figure 2.3. Tensile strength solder joint as a function of aging duration at various temperatures Cu/NiP/Sn3.5Ag [9].](image)
3. Experimental Procedures

This part of the report handles the experimental procedures of the project such as the preparations of the Cu/NiP/SnAgCu solder joints, the thermal aging, the tensile test and the fracture analysis of the solder joints.

3.1 Preparation of Solder Joints
To prepare the solder joints the first step was electroless plating on the substrate. The surface of the copper plates was first polished, and then ultrasonically cleaned with acetone and ethanol, in each solution for 10 minutes. The samples were then etched with nitric acid (HNO₃) for 30 seconds, and then cleaned in de-ionized water. Electroless NiP was plated on the plate by first activate the surface by commercial pre-initiator. Then electroless NiP was plated on the surface using commercial electroless NiP solution. A thin layer of non-cyanide immersion gold was then deposited on the electroless NiP surface to protect the surface from oxidation.

3.2 Joining
A thin layer of no-clean flux was applied to the surface of the NiP coated copper plates. Then the solder wire (SnAgCu) was cut in the same size as the width of the plates. The two plates were fixed in a holder and the solder wire was put between the two plates, the setup design can be seen in figure 3.1.

![Figure 3.1 The specimen setup before being heated.](image1)

To join the samples, the specimen setup was sent into the reflow oven and heated, seen in figure 3.2. After being in the oven the specimen setup was cooled in air.

![Figure 3.2 Specimen setup in the oven.](image2)
3.3 Cutting
Before cutting the samples were molded in epoxy. The as-joined specimens were cut into thin plates with rectangular cross section of the dimension of 12 x 20mm and with a thickness of 0.65 mm, using a precision diamond cutter. The cut plates are shown in figure 3.3.

![Figure 3.3 Cross section of the specimen setup cut into plates.](image)

3.4 Aging
The as-joined specimens were thermally aged at 180° C for 100, 200, 300, 400 and 500 hours. After the thermal aging the samples were cooled in air.

3.5 Tensile Test
The thin plates where then molded in epoxy and cut into rods with the cross-section of 0.65x0.65 mm, using a precision diamond cutter. The design can be seen in figure 3.4. One as-joined specimen was also cut in to rods and prepared for the tensile test. The rods can be seen in figure 3.5.

![Figure 3.4 The design of the rods](image)
Figure 3.5 Samples before tensile test.

The tensile tests were performed using an Instron 5567 tensile tester (Instron, Boston, MA) at room temperature with a constant crosshead speed of 0.05mm/min. Load and extension were recorded during the tests. The maximum load divided by the measured cross-sectional area of the solder joint gave the tensile strength. Three samples were tested from all aging durations. One of the tensile tests is seen in figure 3.6.

Figure 3.6 Tensile test

3.6 Fracture Analyze
The fractured samples were observed by an optical microscope (Olympus BX51) to find out the fracture path. All micrographs are in magnification 5x/0.10 BD.
4. Result & Discussion

In this report an alloy of nickel and phosphorus were plated on copper plates by electroless plating. The plates were joined together using SnAgCu solder. Tensile tests were made to evaluate the mechanical properties of the solder joints. To evaluate its suitability for high temperature applications the samples were aged on a constant temperature of 180°C and at long durations (100, 200, 300, 400 and 500 hours) before the tensile tests were made. In the coming part of the report the results from the tensile tests will be presented and discussed, as well as the micrographs showing the fracture behavior.

4.1 Results

4.1.1 Tensile Test

To evaluate the mechanical properties the ultimate tensile strength (UTS) from the tensile test can be studied. It gives indication of the mechanical strength of the solder joints.

Table 4.1 Mean values of the UTS for three samples from each aging duration.

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<tr>
<th>Aging-time</th>
<th>UTS [MPa]</th>
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<tr>
<td>As-joined</td>
<td>40</td>
</tr>
<tr>
<td>100 hours</td>
<td>39</td>
</tr>
<tr>
<td>200 hours</td>
<td>42</td>
</tr>
<tr>
<td>300 hours</td>
<td>36</td>
</tr>
<tr>
<td>400 hours</td>
<td>31</td>
</tr>
<tr>
<td>500 hours</td>
<td>28</td>
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Table 4.1 shows the UTS of the samples for the different aging durations. For each condition three samples were tested, the table shows the mean value of the three samples. The as-joined samples have a mean value of 40 MPa. A gradual decrease in the UTS is seen at 300 hours of aging (36 MPa), at 500 hours the UTS has decreased to 28 MPa. A plot over the decrease in mechanical strength is shown in Figure 4.1.

Figure 4.1 Mean values of the UTS in MPa as a function of aging duration.
4.1.2 Fracture behavior

The fracture paths were analyzed by micrographs from an optical microscope. One solder joint from each ageing condition is shown in figure 4.2-4.3. The as-joined solder joint, the 100 hours and the 200 hours solder joints have a necking and ductile fracture, seen in figure 4.2 and 4.3 a). The fracture is inside the bulk solder. Sample 300 hours, shown in figure 4.3 b), have some plastic deformation, but the fracture is at the interface between the solder and the substrate. The solder joints aged for 400 hours and 500 hours, seen in figure 4.4, have plastic deformation and the fracture is in the interface.

![Figure 4.2 Micrograph of the fracture path on solder joints after tensile test. a) as-joined and b) 100 hours](image)
Figure 4.3 Micrograph of the fracture path on solder joints after tensile test. a) 200 hours aging and b) 300 hours aging.
Figure 4.4 Micrograph of the fracture path on solder joints after tensile test. a) 400 hours aging and b) 500 hours aging
4.2 Discussion
As mentioned previously in this report it is of greatest concern to use lead-free solders in electronic products because of the health and environmental hazards arising with the use of lead. Lead has been used in solders because of its suitable properties. However, lead is legally prohibited in the European Union and as a consequence the electronic industry is aiming to produce lead free products.

The use of lead free solders causes some problems with the reliability. The reliability of solders in electronics is an important issue for the industry; the failure of a solder joint can destroy the whole function of a product. SnAgCu is a commonly used ternary alloy for solder joints. Compared to lead-rich solders, tin-rich solders react more rapidly with the copper substrate. The reaction results in formation of brittle intermetallic compounds and in poor mechanical reliability. By addition of nickel in the UBM, the interfacial reaction can be slowed down. Electroless nickel-based UBM is used because of its good wettability and low cost.

In this project the objective was to evaluate the mechanical reliability in high temperature applications of SnAgCu solder joints with NiP UBM. Tensile tests were made on the solder joints after thermal aging. From the results, the mechanical reliability in high temperature applications can be discussed and evaluated. To evaluate the mechanical properties one can look at the ultimate tensile strength (UTS). The UTS gives indication of the mechanical strength of the solder joint. The higher the UTS, the more stress the solder joint can stand and the better are the mechanical reliability.

As seen in chapter 4.1.1, in Figure 6 the tensile strength decreases as the aging duration of the samples increases. The as-joined samples have a mean value of the tensile strength of 40 MPa, the 100 hour samples have a mean value of 39 MPa and the 200 hours sample 42 MPa. The tensile strength for the as-joined sample seems low, the samples aged at 100 hours and 200 hours had almost the same result. The mechanical strength should decrease after thermal aging. The reason for the slow decrease in the beginning could be due to slow consumption of the UMB. The decrease continues, at 300 hours (36 MPa), 400 hours (31 MPa) and at 500 hours the tensile strength has dropped to 28 MPa.

From micrographs of the fractured samples, the fracture paths and the fracture behavior can be discussed. The fracture behavior seen in the micrographs (figure 4.2-4.4) shows a result, where the fracture shifts from ductile to brittle at 300 hours aging time. The fracture path also changes at 300 hours, from being inside the bulk solder to being in the solder/substrate interface.
In figure 4.2 and 4.3 a) show the micrographs of the fracture path for the as-joined, 100 hours and 200 hours solder joint. If the fractures presented in chapter 2.4 are being used the fractures could be categorized as ductile fracture inside the bulk solder. The fracture path is inside the solder indicating that the adhesion between the solder, the substrate and the intermetallic layer, and the layer itself is stronger than the strength of the bulk solder and can bear up the applied stress. The necking confirms the ductile behavior. When the solder is deformed necking occurs with increasing plastic strain, and the hardening around the neck results in increased resistance to deformation. As seen the fractured surface is inclined at about 45 deg to the tensile-stress axis.

Figure 4.3 b) and 4.4 a), the solder joints aged for 300 and 400 hours, the fractured surface is uneven. Also, some plastic deformation has occurred, but the ductility is limited. The fracture path is at the interface between the solder and the intermetallic layers. A complete analysis to see if the fracture is trough one or all of the interfacial layers is not possible without analyze of scanning electron microscope (SEM) and energy-dispersive X-ray (EDX). The reason for the weakening could be due to the stress generated between the intermetallic compounds and the solder, the stress is caused by volume mismatch between neighboring layers, the thicker the intermetallic compounds, the larger the stress.

Figure 4.4 b), the solder joint aged for 500 hours, do not have necking and it does not look like the solder has been deformed at all, which should imply a very low ductility. The fracture is between the solder and the intermetallic layers. Cracks are probably due to the stress caused by the volume change during phase transformation in the NiP layer.

The tensile strength decrease when the aging duration increases. The aging duration is equivalent to the time for the reaction of solid-state diffusion and formation of intermetallic compounds. The solder joints aged for longer times has thicker intermetallic compounds, which explains the decrease in mechanical strength. When ductility decreases, the reliability decreases as brittle failures are harder to predict and are more sensitive for cracks because there is no deformation before failure occurs.

Compared to previous research, presented in chapter 2.4, where the same testing method was used on the same substrate and UBM but with Sn3.5Ag solders, the decrease in this report is happening more gradually. In the case with the Sn3.5Ag solder the mechanical strength decreases to low values already at 50 hours of aging duration, and after the UTS is almost constant. In this report the decrease happens gradually, and does not reach a low value until 500 hours aging duration. This could be because of the copper in the solder is leading to a slower consumption of the UBM. Also, the intermetallic compounds formed is probably CuSn instead of the more brittle NiSn. However, this results could just be a trend in this project since a weakness in the experiment is the amount of samples which, to make the results believable, should be higher. Also, analyze with SEM and EDX should be performed to make more trustworthy conclusions regarding the intermetallic compounds.
5. Conclusion & Future Work

5.1 Conclusion
The first conclusion is by increasing the aging duration, decreases in mechanical strength occur. Also, in the ductility a decrease is seen. The fractured samples have necking for the shorter aging durations whereas at longer aging duration no plastic deformation is seen. The decrease is happening gradually, the fracture path however changes from being inside the bulk solder (at 200 hours) to being in the interface (at 300 hours). When the fracture is inside the bulk solder the adhesion for the solder is still good. This makes it possible to conclude that the NiP UMB is not consumed at these ageing durations.

To make the next conclusion the results from the solder used in this report (SnAgCu) are compared to previous research on Sn3.5Ag/NiP solder joints. Both of the solders show decrease in the mechanical properties when thermal aging duration increases. However, the addition of copper makes the decrease in mechanical strength happen more gradually because of a slower consumption of the NiP UMB.

5.2 Recommendations for Future Work
Based on the understanding of the literature and the presented work some recommendations for future work will be presented.

One recommendation is to involve a higher number of samples, to make the results more accurate. Due to lack of time there was not possible in this project to look into the compositions and structure of the intermetallic compounds by SEM and EDX. But this would have been an interesting point and would strengthen the theory what intermetallic compounds are being formed.

As both shear test and tensile test has been made would it also be interesting to look at other aspects for the fracture behavior and reliability, such as cyclical load, to get another fracture.
6. References


2012-04-16


2012-03-09

2012-03-09


