Morphology Analysis of Spontaneous Tin (Sn) Whisker Growth on Lead-Free Solder

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Abstract. Due to the transition of the electronics industry to lead-free solders, the issue of tin (Sn) whisker has become one of the most imperative issues that need to be resolved. Moreover, with the increasing demand for electronics miniaturization, Sn whisker growth is a severe threat to the reliability of microelectronic devices. Sn whisker is a conductive tin structure that grows spontaneously from tin finished surfaces, which can lead to well-documented system failures in electronics industries. Assessment of the Sn whisker growth is difficult because of the slow and unpredictable nature of the Sn whisker formation. This paper presents the results of morphology analysis of Sn whisker growth in terms of Sn whiskers shape, average density and maximum length of Sn whisker on Sn-Cu solder finished using scanning electron microscope (SEM). Micro-indentation test was used to accelerate the growth of Sn whisker at room temperature storage.

1. Introduction
Due to the implementation of the Restriction of Hazardous Substances Directive 2002/95/EC (RoHS) in 2006 prohibiting the use of lead in electronics, the issue of tin (Sn) whisker has become one of the most imperative issues that need to be resolved [1]. Moreover, with the increasing demand for electronics miniaturization, Sn whisker growth is a severe threat...
to the reliability of microelectronic devices [2]. Sn whisker is a conductive tin structure that grows spontaneously from tin finished surfaces, which can lead to well-documented system failures in electronics industries [3-5].

It has been believed that compressive stress in Sn solder coating is a necessary factor for tin whisker growth [6, 7]. Many factors may contribute to the compressive stress level in Sn solder finished, including residual stresses generated during plating process, formation of intermetallic compound (IMC) due to interfacial reaction between Sn solder and copper substrate, mechanical loading, surface damage, and mismatch of coefficient of thermal expansion (CTE) between solder and substrate, or under layer [6].

Whiskers have been observed in a variety of shapes including filaments, nodules and hillocks [8]. Filament whiskers are mostly monocrystalline, needle-like have been observed to grow with lengths up to millimetres but lengths in the 10–100 μm range are observed commonly. Column and spiral whiskers are quite understandable. Nodule whiskers are normally defined by their diameter and are often referred to as mounds or hillocks [5].

Assessment of the Sn whisker growth is difficult because of the slow and unpredictable nature of the Sn whisker formation. It is very hard to estimate the period for the growth of whiskers, which can vary from hours to years. Effects of applied mechanical stress or mechanical deformation on tin whisker growth have been investigated in a few studies. Although a greater extent of mechanical loading or deformation on tin plating could enhance tin whisker growth, little quantitative work has been conducted to correlate the relationship between tin whisker growth and continuously applied mechanical stress [6]. The approach by Doudrick et al. [9] has been successfully used to accelerate whisker growth using microscale indenters. Micro-indentation, is a simple and cost effective method which consist of a ball shaped indenter, has been revealed to induce potential growth of whiskers beyond the directly damaged area and increases the overall stress. As stresses are induced by the IMC and indenter, such as a small ball-bearing, whiskers and hillocks will form [9].

This paper presents the morphology analysis of Sn whisker growth in terms of Sn whiskers shape, average density and maximum length of Sn whisker on Sn-Cu solder finished using scanning electron microscope (SEM) and Image-J Software according to JEDEC standard JESD22-A121. A simple micro-indentation test was used to accelerate continuously the growth of Sn whisker at room temperature storage for 24 h.

2. Methodology
In this experiment, Cu substrates (>99.9% Cu) were used from sheared 1.5 cm x 1.5 cm squares of 1.0 cm in thickness. The Cu substrates were
immersed into acid liquid bath that contain of 5 g (35%) of hydrochloric acid with 95 g of deionized water (1.75%) for 5 s to remove surface oxides, rinsed with acetone followed by distilled water and dried. Flux solution was applied to the Cu substrates before solder-dip process. The flux used was a standard B type (JIS Z3198-4), which is mixtures of rosin, 2-propanol and diethylamine hydrochloride to remove the oxidation from the solder as well as from the surface of the substrates. The Cu substrate was then mounted on the clip holder of a solder-dip machine followed by immersed in the Sn-0.7Cu molten solder bath with withdrawal speeds of 10 mm/s and an immersion time of 5 s using the dipping method. Table I shows the temperature profile parameters of the Sn-Cu solder-dip process. The solder bath is held at a peak temperature of 250 °C. After the solder-dip, the samples were cooled down before the water rinse in ultrasonic bath.

| Solder-Dip Profile Data       | Value |
|-------------------------------|-------|
| Preheat Temperature (°C)      | 200   |
| Preheat Time (s)              | 60    |
| Preheat Rate (°C/s)           | 2.5   |
| Peak Temperature (°C)         | 250   |
| Cooling Rate (°C/s)           | 3.5   |

Micro-indentation test consisted of a 200 g weight was used to accelerate the growth of Sn whisker at room temperature storage. After the samples were indented for 24 hours, morphology analysis of Sn whisker growth in terms of Sn whiskers shape, average density and maximum length of Sn whisker was done using scanning electron microscope (SEM) and image-J Software according to JEDEC standard JESD22-A121.

3. Results and Discussion
Sn whiskers growth on Sn-0.7Cu solder samples were observed after 24 h continuously accelerated with indentation compressive stress. The properties of Sn whiskers were shown in Table 2. The average of Sn whisker length was about 430 μm and the longest Sn whisker was about 920.02 μm. The average density of Sn whiskers was noticed on the surface was about 28 mm⁻² with the average diameters of Sn whiskers were 4.6 μm.
Fig. 1 was showed SEM observations that whiskers formation was greatly enhanced by induced mechanical compressive stress. All whiskers found were filament-like whiskers, hillocks, and nodule-like whiskers. In Fig. 1(b), the long and spiral whisker with length was 294.47 μm apparently grew from a defect on the tin oxide surface. According to Lin and Lin [6], defects, cracks or weak spots, either pre-existent before mechanical testing or induced by mechanical bending, could provide a path for whiskers to initiate and grow for relaxation of the built-up compressive stress. The most well-known as shown in Fig. 1(c) is the filament or needle type whisker: long and straight ones with small diameter. This type

Table-2. The properties of Sn whiskers after 24 h indentation test.

| Sample (indented 48h) | Average whisker length (μm) | Max whisker length (μm) | Average Whisker Diameter (μm) | Whisker Density (mm⁻²) |
|-----------------------|-----------------------------|-------------------------|-------------------------------|------------------------|
| Sn-Cu                 | 430.61                      | 920.02                  | 4.6                           | 28                     |

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Fig. 1. SEM images of the types of Sn whiskers growth on Sn-Cu solder after micro-indentation test at room temperature for 24h. (a) Kinked whisker (b) Spiral whisker (c) Filament whisker

of whisker significantly is the most dangerous to circuit reliability as the conductive tin whiskers can grow long enough to cause electrical shorts circuit by bridging neighbouring conductors or may break off and cause damage elsewhere in the device.

After indentation test, whiskers and hillocks formed around the indentation area. The morphology of Sn whiskers was shown in Fig. 2. Obviously, the hillock and the nodule type of whiskers can be seen a lot in this sample. Even though hillock are short, thick objects protruding not so high from the surface but these types of whisker can be regarded as whisker roots, because if it has the time and conditions, it is likely to grow longer and form other types of Sn whisker [10].
Fig. 2. SEM images of the Sn whiskers morphology at indentation area of Sn-Cu solder (yellow line area showed distribution of hillock (a) Spiral whisker (b) Kinked whisker (c) Filament whisker.

4. Conclusions
From the research, the following conclusions have been obtained:

i. After 24 h continuously indented by mechanical compressive stress, the Sn-0.7Cu samples started extreme whiskering, with the average of Sn whisker length was about 430 μm and the longest Sn whisker was about 920.02 μm.

ii. Sn whiskers nucleate and grow critically after accelerated by micro-indentation test on Sn-Cu solder joint. Sn whiskers found were filament-like whiskers, hillocks, spiral, kink and nodule-like whiskers.

iii. Sn whiskers significantly is the most dangerous to circuit reliability as the conductive Sn whiskers can grow to several hundred microns, which are long enough to cause electrical shorts circuit by bridging neighbouring conductors or may break off and cause damage elsewhere in the device.

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