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Plastic Deformation Effect on Sn Whisker Growth in Electroplated Sn and Sn-Ag Solders

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Abstract

Sn whiskers are becoming a serious reliability problem in microelectronics where Pb-free solder technology is being implemented with pure Sn or Sn-rich alloys. Numerous investigations have been performed to understand the whisker growth mechanisms and thereby to mitigate Sn whisker growth. Among many Sn whisker mitigation strategies, minor alloying additions to Sn have been found to be quite effective. One challenge in evaluating Sn whisker growth is a time-consuming aging test, such as 4000 h testing condition recommended by the JEDEC standard.

In this study, several commercial Sn and Sn-Ag baths of low whisker formulations are evaluated. The effects of plating variables and aging conditions on Sn whisker growth are investigated with matte Sn, matte Sn-Ag, and bright Sn-Ag electroplated on a Cu/Ni/Si substrate. The layer thickness and current density are the major plating variables studied. Two different storage conditions are applied; an ambient condition (30°C/dry air), and a high temperature/humidity condition (JEDEC, 55°C/85%RH).

In addition, the effect of plastic deformation on Sn whisker growth is investigated as an acceleration method for Sn whisker testing. Microhardness indentation technique is applied to electroplated Sn and Sn-Ag samples to plastically deform them before T/H testing. Each sample is examined by SEM at a regular time interval up to 4000 h. Various morphologies of Sn whiskers are observed and their growth statistics are analyzed in terms of plating conditions, plastic deformation and T/H testing conditions. Plastic deformation is found to significantly accelerate Sn whisker growth both in pure Sn and Sn-Ag samples. The method of plastic deformation can be employed to shorten the time-consuming Sn whisker growth testing.

Introduction

Sn whiskers are electrically conductive crystalline structures formed on Sn-rich surfaces. Their typical dimensions are; 10 to 1000 μm in length and 0.1 to 10 μm in diameter. A typical growth rate ranges from 30 to 9000 μm per year. Sn whiskers are commonly found on electroplated materials used in aerospace, automotive, and electronics applications [1,2]. Several failure types associated with Sn whiskers have been reported such as electrical short circuit, contamination, metal vapor arc, and others [3]. Sn whiskers are becoming a serious reliability issue since Pb-free, Sn-rich solders are replacing Pb-containing solders in electrical and electronic products according to the RoHS legislation [4].

The formation of Sn whiskers has been extensively investigated and numerous growth mechanisms have been proposed [5,6]. However, the growth mechanisms are not completely understood yet. But it is widely understood that compressive stress developed in Sn-rich materials is a necessary factor to promote Sn whisker formation/growth. The compressive stress can be originated from various sources such as residual stresses due to Sn plating process [7], intermetallic compounds (IMCs) growth between Sn and Cu substrates [8], externally applied stresses [9], coefficient of thermal expansion (CTE) mismatch [10], and others. Accordingly, many mitigation strategies of Sn whiskers have been practiced to reduce compress stresses, to prevent intermetallic formation, or to minimize thermal expansion mismatch [11,12].

In this study, the plating variables of matte Sn, matte Sn-Ag and bright Sn-Ag baths (of low whisker formulations) are investigated to establish optimum mitigation strategies for Sn whisker growth. The effects of Ag addition and storage conditions are also evaluated. In addition, the effect of externally applied stress/strain is investigated to accelerate Sn whisker growth by comparing plastically deformed samples with no-deformation samples in JEDEC storage conditions. The plastic deformation experiment is designed in an effort to shorten the time-consuming JEDEC tests.

Experimental procedures

Commercially available matte Sn, bright Sn-Ag (from vendor A), and matte Sn-Ag (from vendor B) low-whisker formulations were used for electroplating. The Ag contents of matte Sn-Ag, and bright Sn-Ag are 2~3, and 4~5 wt%, respectively. Matte Sn and bright Sn-Ag were electroplated using a DC power supply with a DSA inert anode in a small scale bath which has 250 ml capacity. As a substrate, the metallization of TiW(0.16 μm)/Ni(2.4 μm)/Cu(0.8 μm) is deposited by CVD on a 750 μm Si wafer. Matte Sn samples were electroplated at a current density of 10 and 25 mA/cm^2 at 40°C to produce two different plating thicknesses; 2 and 10 μm. In case of bright Sn-Ag, samples were electroplated to 10 μm in thickness by using 75 mA/cm^2 at 27°C. On the other hand, matte Sn-Ag were electroplated to ~10 μm in thickness by using 25 mA/cm^2 after 1 μm Cu pre-deposition on the TiW(1.65 μm)/Cu(4.4 μm)/Cr-Cu substrate.

To study the effect of plastic deformation, 100 gf load is applied to electroplated surfaces using a micro-hardness tester equipped with an indenter having a 136° Vickers diamond square. Deformation speed, and dwell time are 50 µm/sec, and 5 sec, respectively. Four indentation marks are applied in each sample having an area of 1 x 1 cm^2. And the distance between each indentation is more than 500 µm to prevent interference.

Table 1 shows sample matrix in this study. Each group of samples was stored in both the ambient (30°C; dry air) and high temperature and high humidity (55°C; relative humidity: 85%) conditions as per the JESD22A121 standard. Samples were examined every 200 hours up to 1000 hours, then at every 1000 hours until 4000 hours by using scanning electron microscope (SEM). The samples were then sectioned by focused ion beam (FIB) (Nova230; FEI Co., Hilsboro, OR) and the sectioned sides were examined by FIB ion channeling imaging.

Table 1. Sample matrix in this study.

Material	Current density (mA/cm^2)	Plating thickness (µm)
Matte Sn (vendor A)	10	Thin (~2 µm)
		Thick (~10 µm)
	25	Thin (~2 µm)
		Thick (~10 µm)
Matte Sn-Ag (vendor B)	25	Thick (~10 µm)
Bright Sn-Ag (vendor A)	75	Thick (~10 µm)

Results and Discussion

Effects of Ag addition, plating variables, and storage conditions on Sn whisker growth

Figure 1 shows the surface SEM images of as-plated matte Sn, matte Sn-Ag, and bright Sn-Ag with 10 µm plating thickness. Well-polygonized and large grains are observed on the surface of matte Sn regardless of the current density. And the grain diameter is similar for two samples electroplated with 10 and 25 mA/cm^2 current density. By the addition of Ag, the grain diameter of matte Sn is significantly reduced from 4.5 to 1.5 µm, which is consistent with the previous work [13]. The surface of matte Sn became much more smoother [14]. On the other hand, the grain diameter of bright Sn-Ag was significantly reduced to less than 50 nm, possibly due to Ag brightener. [15]

The effect of Ag addition on Sn whisker growth is investigated. Figure 2 shows the SEM images of Sn whiskers on the surfaces of electroplated matte Sn, matte Sn-Ag, and bright Sn-Ag stored at an ambient condition for 4000 h. Sn whiskers are observed only in matte Sn surface, while no Sn whiskers are observed both in matte Sn-Ag and bright Sn-Ag. As shown in figure 3, eighty (80) Sn whiskers are observed per mm^2 in matte Sn surface, and this result greatly exceeds the JEDEC criterion of fortyfive (45) Sn whiskers. And the maximum Sn whisker length observed on matte Sn is about 96 µm. On the other hand, Sn whisker is not observed at all on matte Sn-Ag, and bright Sn-Ag. Bright Sn is generally known to have a larger Sn whisker density and longer maximum length than matte Sn due to the organic additives. [15,16] However, in this study, no Sn whiskers are observed on both matte Sn-Ag and bright Sn-Ag due to addition of Ag.

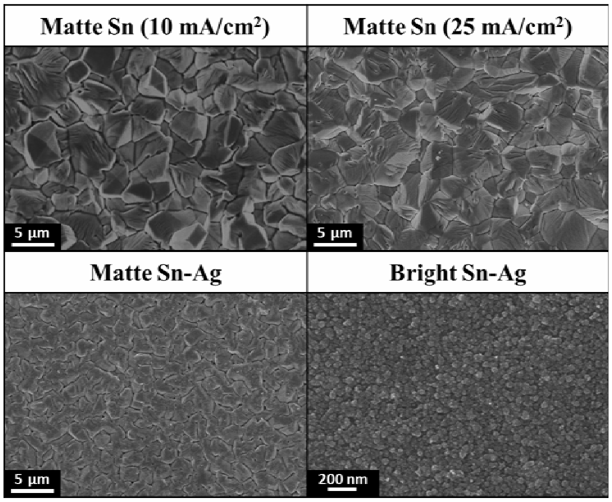
Matte Sn (10 mA/cm^2)
Matte Sn (25 mA/cm^2)
Matte Sn-Ag
Bright Sn-Ag

Figure 1. SEM images of as-plated surfaces of electroplated matte Sn, matte Sn-Ag, and bright Sn-Ag with 10 µm plating thickness.

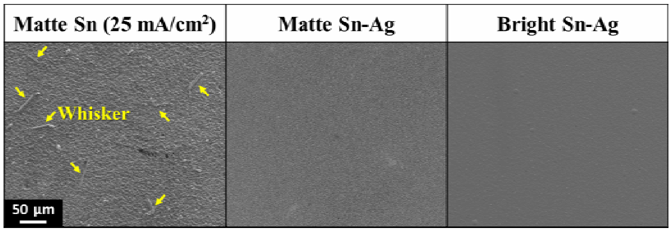
Matte Sn (25 mA/cm^2)
Matte Sn-Ag
Bright Sn-Ag

Figure 2. SEM images of the surfaces of matte Sn, matte Sn-Ag, and bright Sn-Ag with 10 µm plating thickness stored at an ambient condition after 4000 h. Sn whiskers are only observed on matte Sn.

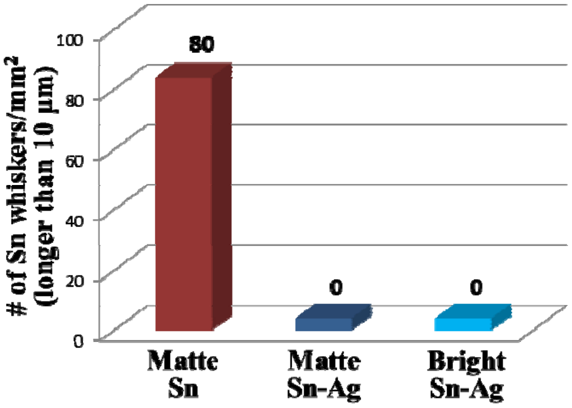

Figure 3. Sn whisker density of matte Sn, matte Sn-Ag, and bright Sn-Ag with 10 µm plating thickness samples stored at an ambient condition after 4000 h.

The effects of plating variables, current density and plating thickness, on Sn whisker growth are investigated in matte Sn. All matte Sn samples stored at an ambient condition have Sn whiskers after 4000 h as shown in Figure 2. There are three types of Sn whiskers; straight, kink/bend, and nodule type. Straight and kink/bend type whiskers can grow longer than a few millimeters, while nodule type cannot grow long as straight and kink/bend types. Therefore, straight and kink/bend types would be more concerned in the reliability issue than nodule type. Figure 4 shows the number of Sn whiskers which are longer than 10 μm on the surfaces of matte Sn stored at an ambient condition after 4000 h. Thin matte Sn electroplated at the high current density has the largest Sn whisker density. Matte Sn at the low current density has a similar density of 59 whiskers/mm^2 regardless of plating thickness. In matte Sn with the high current density, the number of large whiskers dramatically decreases as the plating thickness increases. Nodule type whiskers are the majority in thick matte Sn regardless of current density. According to the whisker density range of JEDEC JESD22-A121A, all matte Sn samples stored at an ambient condition show a higher number of whiskers per area (mm^2) than the allowable number of 45 whiskers.

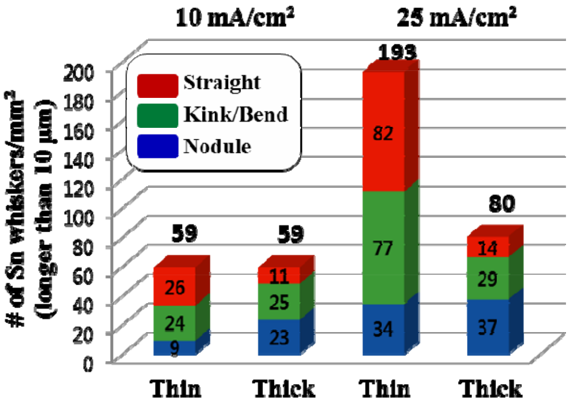

Figure 4. Sn whisker density of matte Sn using different current density with different plating thickness samples stored at an ambient condition after 4000 h.

The length of Sn whiskers is also investigated by various whisker types. Figure 5 shows the average length of Sn whiskers which is longer than 10 μm on the surfaces of matte Sn stored at an ambient condition after 4000 h. Kink/bend type whiskers have the longest average and maximum length. Nodule type whiskers have the shortest length as about 10 μm. Thick matte Sn samples have much longer Sn whisker than thin matte Sn. The maximum lengths of Sn whiskers are 52, 89, 90, and 96 μm for 10 mA/cm^2 (thin), 10 mA/cm^2 (thick), 25 mA/cm^2 (thin), and 25 mA/cm^2 (thick), respectively. According to JEDEC JESD 291A, the maximum allowable Sn whisker length is 40 μm for automotive and 50 μm for consumer products. The maximum length of Sn whiskers on the matte Sn stored at an ambient condition does not satisfy the JEDEC criteria regardless of plating variables.

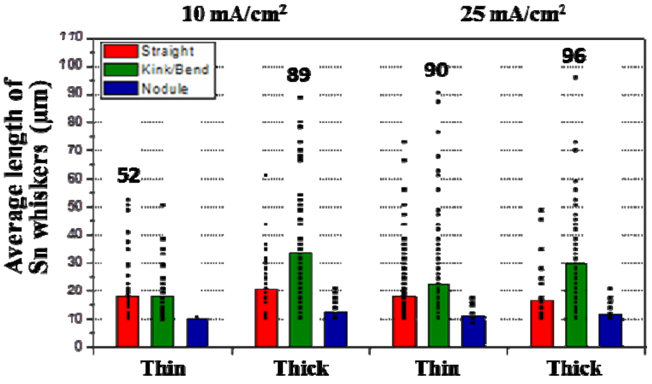

Figure 5. Average length of Sn whisker in matte Sn using different current density with different plating thickness samples stored at an ambient condition after 4000 h.

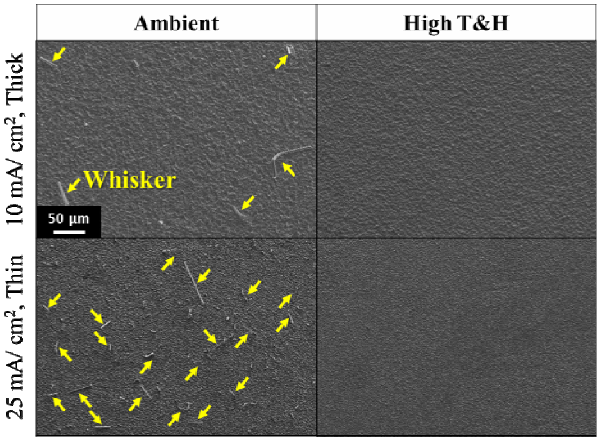

Figure 6. SEM images of surface on matte Sn stored at an ambient and a high temperature/humidity condition after 4000 h.

It is interesting to note that, no Sn whiskers are observed on the matte Sn stored at a high temp/humidity condition as shown in figure 6. To understand the effects of high temperature and humidity, in-depth analysis was performed on the microstructural changes and surface oxidation. Figure 7 shows the ion channeling images of cross-sectioned matte Sn electroplated at 25 mA/cm^2 and stored after 4000 h. Cu_6Sn_5 grows dominantly along Sn grain boundaries in matte Sn stored at an ambient condition, while Cu_6Sn_5 and Cu_3Sn grow randomly in matte Sn stored at a high temp/humidity condition. Randomly-grown-IMCs may better suppress Sn whisker growth because they can more effectively reduce the stress concentration within grains as well as grain boundaries. The suppression of Sn whisker growth in high humidity can also be understood in term of the surface oxide thickness. Figure 8 shows TEM images of 10 μm thick matte Sn electroplated using 25 mA/cm^2 stored after 3000 h. Thick oxide layers are observed on the surfaces of matte Sn stored at a high temp/ humidity condition whereas a thin oxide (only native oxide layers) is observed in the surfaces of matte Sn stored at an ambient condition. Therefore, Sn whiskers may be difficult to form on the surface of matte Sn stored at a high temp/ humidity condition probably due to a thicker oxide layer.

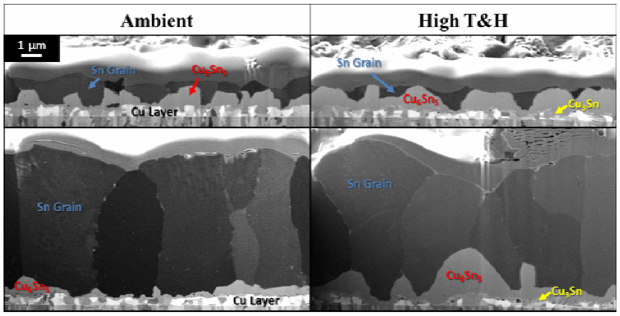

Figure 7. Ion channeling images of cross-sectioned matte Sn, electroplated using 25 mA/cm^2, stored at an ambient and a high temperature/humidity condition after 4000 h.

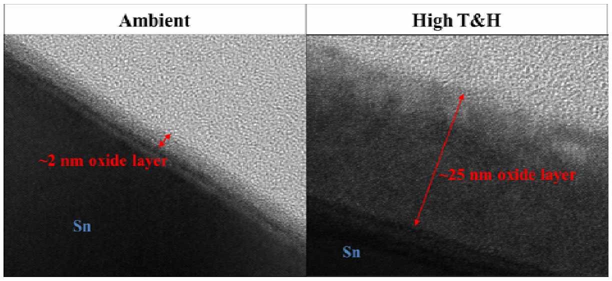

Figure 8. TEM images of 10 µm thick matte Sn, electroplated using 25 mA/cm^2, stored at an ambient and a high temperature/humidity condition after 3000 h. A much thicker oxide layer is observed for the sample stored at the high temperature/humidity condition.

Effects of plastic deformation on Sn whisker growth

Surfaces of plastically deformed samples are examined by SEM, stored after 4000 h as shown in Figure 9. In matte Sn stored at an ambient condition, Sn whiskers are observed not only in the indented area, but also, outside of the indentation area. However, Sn whiskers are observed only in the indentation area for samples stored at a high temperature/ humidity condition. No Sn whiskers are observed on the surfaces of matte Sn-Ag, while Sn whiskers are observed only in the indented area for both thin and thick samples regardless of the storage condition in bright Sn-Ag.

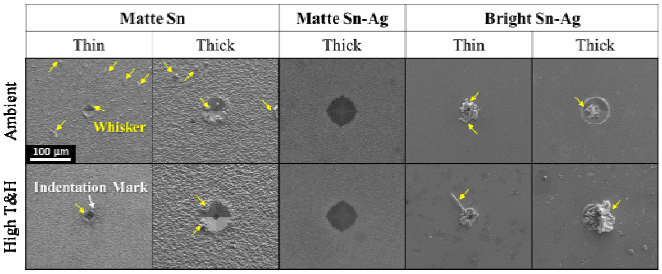

Figure 9. SEM images of plastically deformed surfaces of matte Sn, matte Sn-Ag, and bright Sn-Ag stored at an ambient and a high temp/humidity condition after 4000 h.

Table 2 shows the effect of plastic deformation on Sn whisker growth. In case of no-deformation, Sn whiskers are only observed in matte Sn samples stored at an ambient condition. However, when plastic deformation is applied, Sn whiskers are observed in the indented area which is plastically deformed area. The Sn whisker density and length are compared between plastically deformed samples and no-deformation samples. Sn whisker density of plastically deformed samples of the matte Sn is similar to that of no-deformation samples in the area outside of indentation marks. In the area outside of indentation marks, changes of Sn whisker density with storage time have the similar trends on both plastically deformed and no-deformation samples. Therefore, it is concluded that plastic deformation appear to have no effect in the area outside of indentation marks.

Table 2. Sn whisker growth between plastically deformed samples and no-deformation samples.

Material	Plastic deformation		No deformation	
	Ambient	High T&H	Ambient	High T&H
Matte Sn	**surface area** + **deformed area**	**deformed area**	**surface area**	**no whisker**
Matte Sn-Ag	**no whisker**	**no whisker**	**no whisker**	**no whisker**
Bright Sn-Ag	**deformed area**	**deformed area**	**no whisker**	**no whisker**

Sn whisker growth behavior on the indentation in matte Sn, and bright Sn-Ag is also investigated. Figure 10 shows SEM images of Sn whiskers on the indentation stored at an ambient condition after 4000 h. Sn whiskers are observed on the indentation area at the every condition investigated. Sn whiskers on the indentation are classified as ‘linear’ and ‘nodule’ type. Linear type can be observed only in thin plating samples, not in thick plating samples.

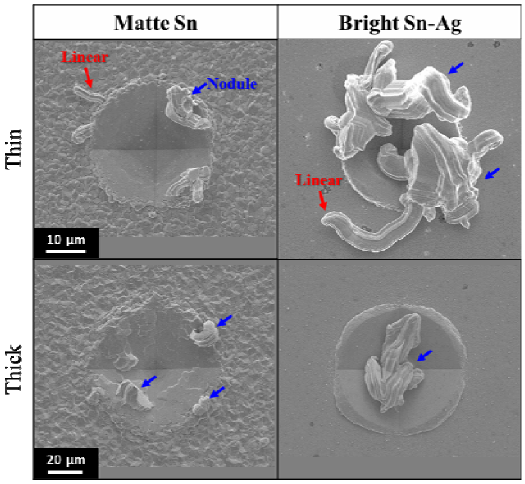

Figure 10. SEM images of Sn whiskers on the indentation in matte Sn and bright Sn-Ag with different thickness samples stored at an ambient condition after 4000 h.

Figure 11 shows the number of Sn whiskers on the indentation of matte Sn, and bright Sn-Ag, stored at an ambient condition after 4000 h. Matte Sn with thin plating sample has the highest Sn whisker density, nine (9.0) whiskers per indentation. Thin plating samples have a higher

Sn whisker density than thick plating samples. And there are no linear type Sn whiskers observed on the indentation of thick plating samples. Figure 12 shows the Sn whisker density changes with storage time. In matte Sn (thin samples), Sn whiskers formation was ended well before 1000 h, while, Sn whiskers grow continuously after 1000 h in the other samples. And thick plating samples have a longer time to form Sn whiskers than thin plating samples.

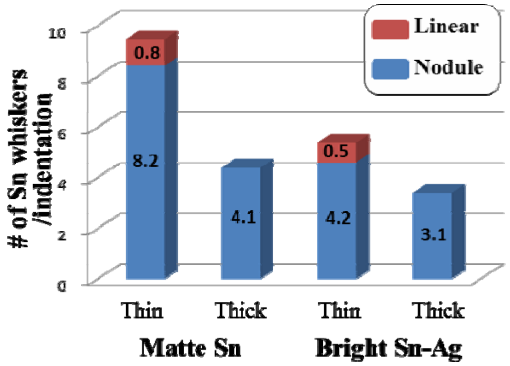

Figure 11. Number of Sn whiskers on the indentation in matte Sn and bright Sn-Ag with different thickness samples stored at an ambient condition after 4000 h.

The length of Sn whisker is also investigated by two whisker types. In matte Sn, nodule type whiskers have the longer length than the linear type, and thick matte Sn has much longer Sn nodules than thin matte Sn does. The length of Sn whiskers on the indentation is shorter than the length of Sn whiskers on on-deformation samples. On the other hand, the maximum length of Sn whiskers is similar in thin and thick bright Sn-Ag, about 30 μm. The longest Sn nodules observed in thin bright Sn-Ag is about 31.9 μm.

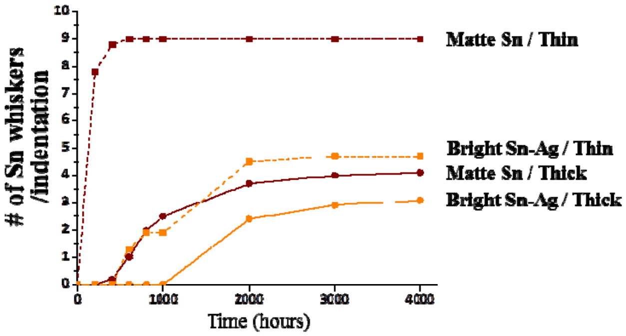

Figure 12. Number of Sn whiskers on the indentation in matte Sn and bright Sn-Ag with different thickness samples stored at an ambient condition with storage time.

Figure 13 shows the schematic diagrams of Sn whisker location on the indentation. In matte Sn, most Sn whiskers are formed along the indentation boundary in thin plating samples, while Sn whiskers are observed mostly inside the indentation (along the diamond-shape boundary) in thick samples. Linear-type whiskers are mostly formed near the circular boundaries. As similar as matte Sn, most of Sn nodules are formed along the indentation boundaries in thin samples, while Sn nodules are observed mostly inside the indentation in thick samples.

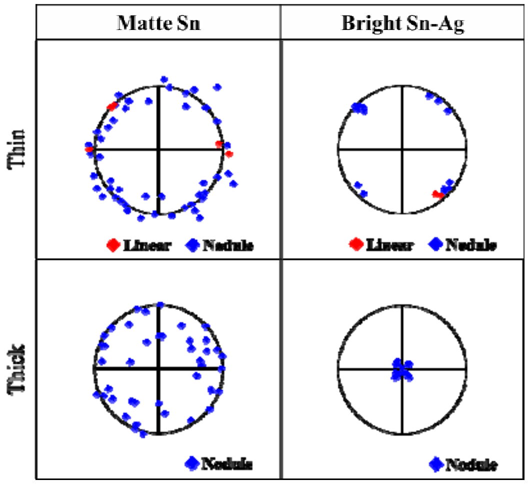

Figure 13. Schematic diagrams of Sn whiskers location on the indentation in matte Sn and bright Sn-Ag with different thickness samples stored at an ambient condition after 4000h.

Summary

By evaluating the important plating variables of several commercial baths of low whisker formulations (including matte Sn, mstte Sn-Ag, and bright Sn-Ag), it is found that control of the plating variables is not enough to suppress Sn whisker growth.

The Ag addition to matte Sn is found to be the most effective mitigation method to suppress Sn whisker growth among others.

Plastic deformation is found to significantly accelerate the formation/growth of Sn whiskers on the indentation marks in both Sn and Sn-Ag samples. Sn whiskers are observed as early after 100 h T/H exposure on plastically deformed Sn-Ag samples.

The method of plastic deformation can be employed to shorten the time-consuming Sn whisker growth testing.

Acknowledgments

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