

Comparison of Whisker Growth from Pressure-Induced and Environment-Induced Whisker Growth Test Methods

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Abstract

One of the remaining challenges associated with the use of lead-free materials in electronics is the potential failure risk associated with tin whiskers. Despite intense research in recent years, there are still no accelerated test methods to reliably predict whisker growth in field application conditions. Semiconductor component and electronic system manufacturers rely on a set of environmental exposure conditions defined by industry standards to qualify and monitor plating processes. While these test methods offer a common platform for package qualification, their value for quantitative whisker-induced failure risk assessment is quite limited. Additionally, these tests are time-consuming and labor intensive, making it difficult to use for purposes such as process improvement, control, and monitoring.

In this paper, a mechanical indentation method is evaluated for assessing the whisker growth propensity of tin and select tin alloys plated on copper, brass, and alloy 42 substrates. Indentation-induced growth results are compared with responses to standard test conditions including temperature cycling and elevated temperature and humidity.

Introduction

Tin whiskers are spontaneous growths that occur on tin and high-tin-content alloys that are used as protective solderable surface finishes. Whiskers can vary significantly in dimensions, ranging from less than a micron to over ten microns in diameter and from a few microns to over one thousand microns in length. Tin whiskers longer than 15 mm have been documented [1]. Since metal whiskers can carry electrical current, their formation presents a failure risk for electronic equipment. Historically, tin whiskers have been blamed for costly system failures. Alloying lead (Pb) with tin has been demonstrated to significantly reduce whisker growth [2]. However, the ban on lead introduced for the majority of electronic equipment in 2006 [3] has raised concerns that electronics may face an increased risk of failure from tin whiskers. To address these concerns, standards have been developed to provide a common set of tests to evaluate whisker growth propensity. These standards include JEITA ET-7410[4], JESD201[5], and IEC 60068-2-82[6]. These standards define three environmental exposures, including two constant temperature/humidity conditions and one temperature cycling condition. The growth propensity tests require up to 3000 hours, not counting inspection time. Tests have been adopted by a number of part assemblers and

part manufacturers to assure customers that parts are not prone to whisker growth.

For equipment manufacturers, it would be desirable to conduct a quick test for evaluating tin whisker formation to assess whisker propensity instead of having long environmental tests. Application of pressure has been known to contribute significantly to whisker growth for decades [7] and has been the subject of recent study [8]-[10]. This paper examines the use of an indentation test to assess the whisker growth propensity of a selected finish. Comparisons of growth by indentation versus standard environmental exposures are presented. This study examines the impact of variations in the plating process and variations on substrate in the ability of indentation to determine whisker propensity.

Test Specimens

Twelve sample conditions were used for this study. A sample condition is identified by the electroplating bath composition, current density, and substrate. Two different Sn baths, one methane sulfonic acid (MSA), and the other based on sulfuric acid, were examined. For the MSA Sn bath, copper substrates were plated at two current densities with and without the

addition of trace amounts of copper (Cu) and lead (Pb). In order to examine impact of plating substrate, a

Table 1 Summary of Conditions Used for the Test

Electroplating	Supplier	Current Density	Substrate	Plating Efficiency	Bath Temp	Plating Thickness	Alloying Composition
Sn (1)	Supplier 1 (MSA baths)	60 mA/cm ²	Cu	>90%	23°C	7-8um	-
SnCu				>90%	23°C	7-8um	0.7% Cu
SnCuPb		120 mA/cm ²		>90%	23°C	7-8um	0.7% Cu, 1.3% Pb
Sn (2)	Supplier 2 (sulfuric acid bath)	25 mA/cm ²	Cu Brass Alloy 42	80%	23°C	7-9um	-
Sn-Bi	Supplier 3	150 mA/cm ²	Cu Brass Alloy 42	>90%	50°C	7-9um	2% Bi

commercial grade Sn-Bi bath and a sulfuric acid Sn bath were used to plate copper (C11000, 99.9% Cu), brass (C26000, 70% Cu, 30% Zn) and alloy 42 (58% Fe, 42% Ni). A summary of the sample conditions can be found in Table 1.

For samples prepared with Supplier 1 ingredients, copper substrates were plated in 1L MSA-based baths at two possible current densities: 60mA/cm² or 120mA/cm². The Sn-Cu and Sn-Cu-Pb baths were prepared by the addition of Cu and Pb into the solution, with a pure tin anode. Prior to plating, substrates were etched in phosphoric acid and rinsed with ultra-pure water. Substrates were then attached to a rotating cathode that spun at 100 rpm for all 60mA/cm² plating, and 200rpm for all 120mA/cm² deposits. Plating was conducted at laboratory-ambient conditions. After plating, the samples were rinsed in ultra-pure water and air dried. All samples had a dull luster to them and fine grain structure.

Samples plated with Supplier 2 electrolytes were polished with Silicon Carbide paper, etched in 25% sulfuric acid, rinsed in ultra-pure water, and then plated in a 0.5L bath. The plating samples were prepared one at a time with the sample (cathode) and pure tin anode positioned vertically in the bath, with a 10 cm separation. The bath was agitated by a rotating bead at 60Hz. Plating was conducted at laboratory-ambient conditions. During plating, hydrogen evolution was observed, which was reflected in lower plating efficiency. After plating, the samples were rinsed with ultra-pure water and air dried. The final finish had extremely small, sub-micron grains, independent of the substrate.

The Supplier 3 Sn-Bi bath was a 0.5L solution agitated by a rotating bead at 60Hz. The bath temperature was maintained at 50°C using a heating plate. Substrates were first polished with silicon carbide

paper, etched in 25% sulfuric acid, rinsed in ultra-pure water, and then positioned vertically in the bath 10 cm from a pure tin anode. The Sn-Bi plating was a high-speed process that deposited a 7-9 µm layer in approximately 75 sec. The Sn-Bi bath was maintained at 50°C throughout the deposition process. No hydrogen evolution was observed during plating. After plating, the samples were rinsed with ultra-pure water and air dried. The deposit had large grains irrespective of the type of substrate, commonly with a diameter of 2-6 µm.

After plating, the thicknesses of all the samples were measured with X-Ray Fluorescence (XRF). The texture of the plating samples is depicted in Figure 1.

Test Conditions

In this study, an indentation test was examined as a method for identifying whisker growth potential. For comparison, the plated samples subjected to the indentation test were also subjected to temperature cycling and elevated temperature and humidity.

The indentation test was conducted by placing a spherical tip indenter on the surface of the plated sample coupons under a 200 gram load for 30 minutes. The stylus was constructed of ruby and had a diameter of 1 mm. Figure 3 depicts the indentation test fixture. The ruby-tipped indenter is depicted in Figure 2. The area around the indentation point was inspected under a scanning electron microscope (SEM) before and after application of the indentation load. An example of an indentation area is depicted in Figure 4. Whiskers were defined to be cylindrical growths with striations having a length-to-diameter ratio of at least 2 to 1. Four indentation points were created per sample. After indentation, separate sets of samples of each type were placed in either temperature cycling or elevated temperature humidity conditions.

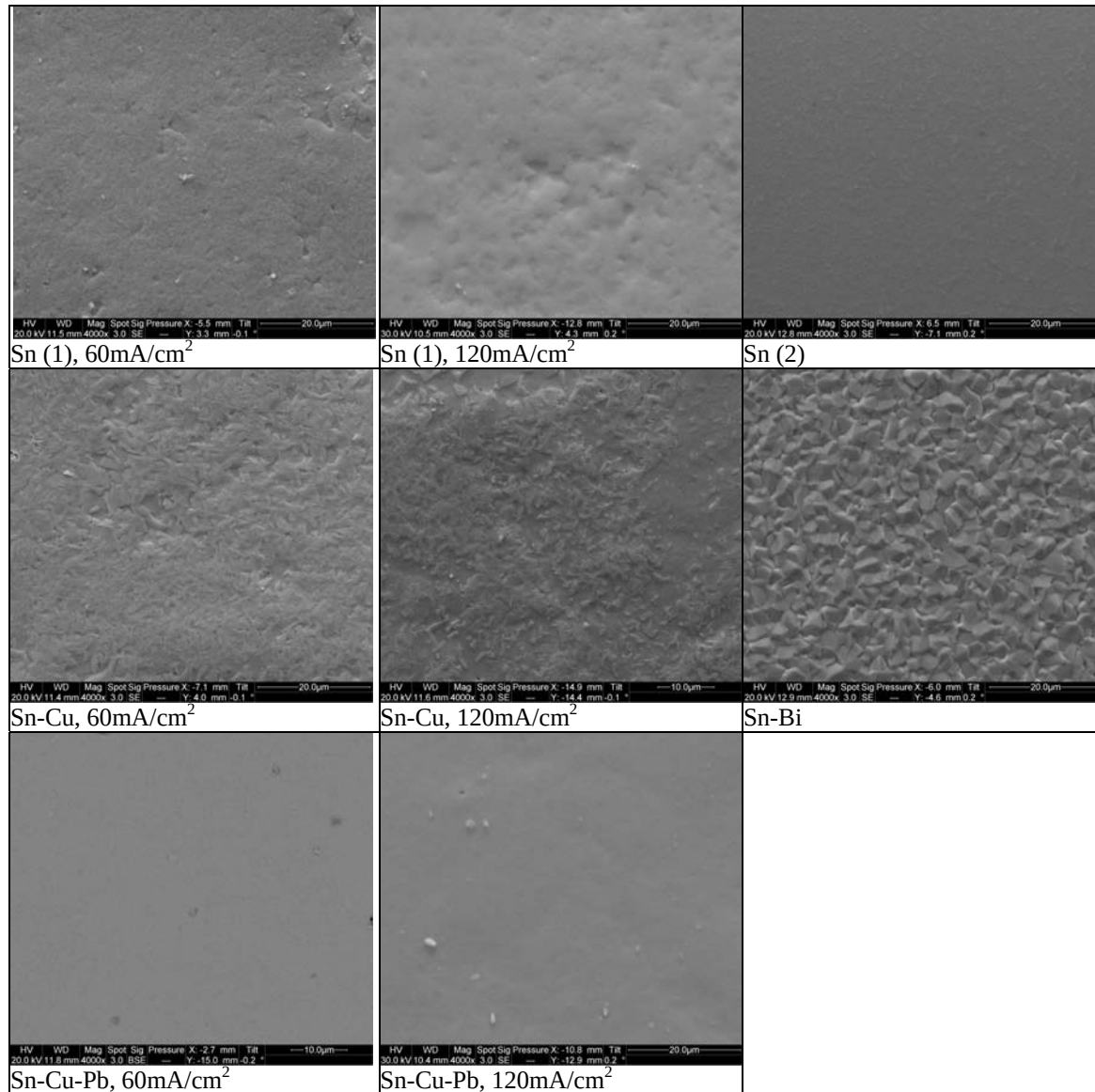


Figure 1 Surface texture of sample conditions

The temperature cycling test was conducted in a two zone oven with a cold temperature zone set at -55°C and a hot temperature zone offset at 85°C . Samples alternated between the hot and cold sides at a frequency of approximately three cycles per hour. For the test, samples were subjected to 1000 cycles with a dwell time of 10 minutes in both the cold and hot temperature zones.

The elevated temperature and humidity test was conducted in an environmental chamber that was held at 55°C and 85% RH. Samples were kept in the chamber for 1000 hours. Both the temperature cycling and the elevated temperature humidity conditions were taken from JESD22-A121 [11] and IEC60068-2-82 [6].

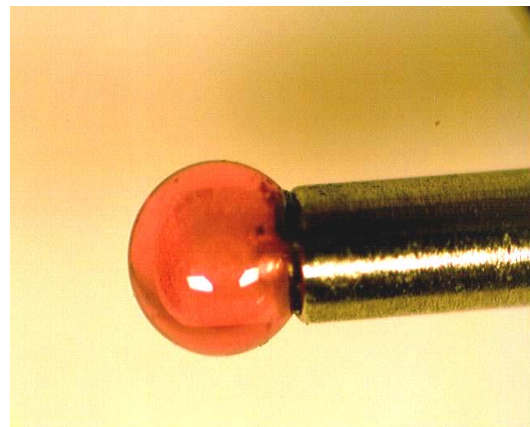


Figure 2 Indenter tip: 1 mm diameter

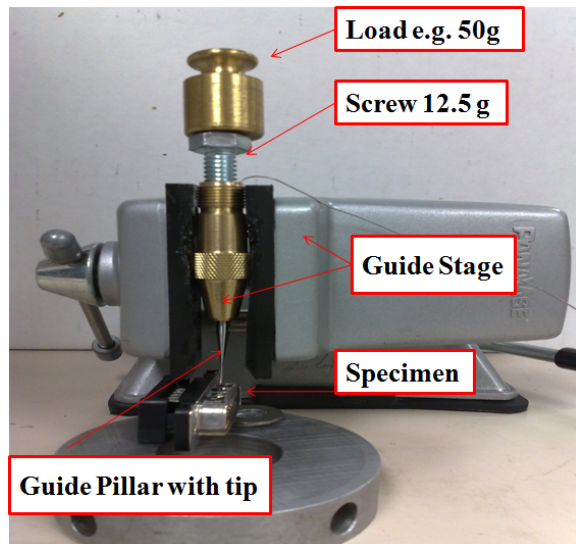


Figure 3 Indentation fixture

Inspection for whisker growth was conducted after environmental exposure. Inspection methods included a surface scan under SEM at 1000X to identify any whiskers. After surface scan, 12 randomly selected areas were examined for whisker growth. Whisker growth measurements included whisker density and length. When available, a maximum of 50 length measurements were taken per sample. To differentiate from indentation induced whiskers, the 12 areas did not include the indentation sites. Indentation sites were inspected separately after environmental exposure.

Preliminary Inspection

A surface scan was conducted on each test specimen prior to indentation and environmental exposure. The samples were checked for the presence of whiskers and/or nodules in order to provide a baseline for whisker results obtained after indentation and environmental exposure tests. The samples were

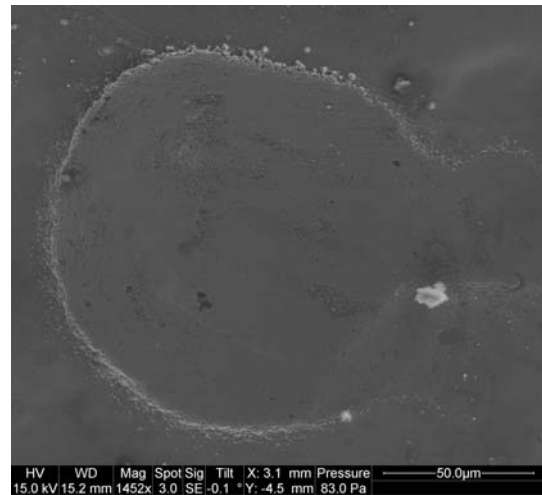


Figure 4 Example of indentation area

2-4 weeks old at the time of the preliminary observations; after the inspections were complete, the samples went directly into test. A summary of preliminary observations along with the test results is provided in Table 2.

Only the Sn-Cu and Sn-Cu-Pb deposits over Cu from Supplier 1 had produced whisker growth prior to the test. For both finishes, nodular and whisker growth was noted: whiskers were differentiated from nodular growth by having a length-to-diameter ratio exceeding 2 to 1. Examples of these whiskers can be seen in Figure 5 and Figure 6. On Sn-Cu-Pb film, some whiskers were not made of pure Sn, but had inclusions of Pb. The lighter-colored inclusions of the whisker in Figure 6 are composed of higher-atomic number Pb as seen under a back-scattering detector using SEM. Such bi-metallic Sn-Pb whiskers have been observed previously through high current density [12] and pressure inducement [13].

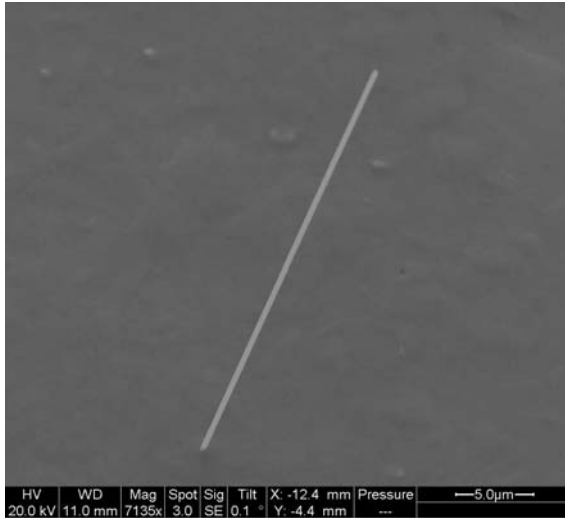


Figure 5 Whisker on Sn-Cu (120mA/cm²) prior to start of the indentation test

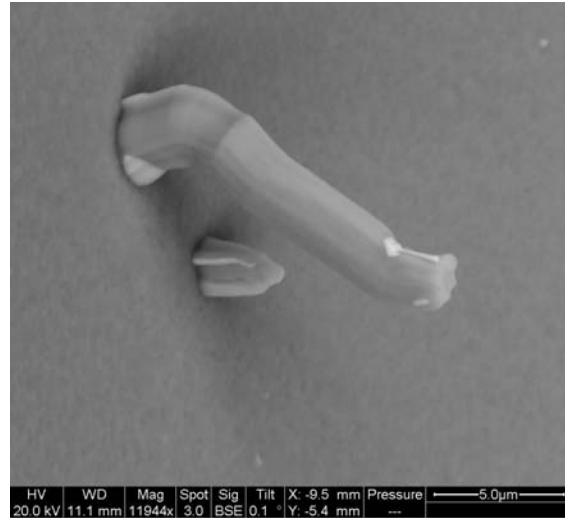


Figure 6 Whisker on Sn-Cu-Pb (60 mA/cm²) prior to start of the indentation test



Figure 7 Indentation site (left) and whisker (right) found on Sn-Cu (60mA/cm²) over copper after 200g indentation test

Indentation test

The indentation test sites were examined under SEM. Whisker formation was documented on the perimeter of each indentation site. Figure 7 depicts an indentation site (left) and a formed whisker (right). The number and lengths of whiskers observed were documented as part of the inspection.

From inspections, all MSA-based plating solutions—Sn(1), Sn-Cu, and Sn-Cu-Pb—were found to produced whiskers. Sn-Bi plating over alloy 42 produced one whisker. All other plating and substrate combinations remained whisker-free. The results are summarized in Table 2 and Table 3.

Temperature cycling and temperature humidity results

For the temperature cycling test, twelve regions with an area of approximately 0.229 mm² were examined at the end of 1000 cycles. In each area, whiskers were counted and the lengths of the five longest whiskers were documented. For MSA-based baths, Sn-Cu and Sn-Cu-Pb were found to have whiskers, while pure Sn(1) did not. None of the Sn(2)-finished samples, regardless of substrate, produced whiskers. For Sn-Bi finish, alloy 42 and brass substrates have shown whisker growth. All results are summarized in Table 2 and Table 3.

It must be noted that whisker growth on SnBi over alloy 42 after temperature cycling was one of the densest ever reported: 4019 whiskers/mm². An

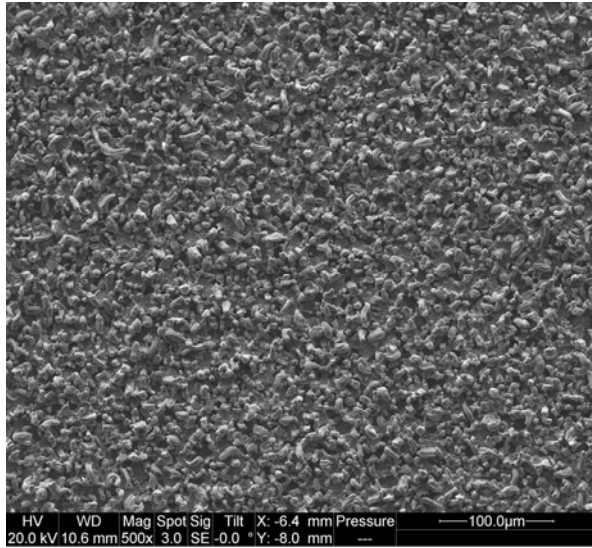


Figure 8 Example of Sn-Bi over alloy 42 surface after 1000 temperature cycles

example of this high density can be observed in Figure 8 and Figure 9. It appears that a large number of the grains became dislodged from their neighboring grains and created whiskers. For grain sizes of 2-6 μm, we can approximate about 50,000 Sn grains per 1 mm². This puts the occurrence of whiskering grains at 1 in every 12-13 grains. Most of the published data puts whisker occurrence as one in every 1,000–10,000 grains.

Results from 1000 hours of temperature humidity exposure (55°C/85%RH) do not mimic the same whisker growth of Sn-Bi over alloy 42, as was



Figure 9 A closer look at Figure 8

seen during temperature cycling. However, growth was observed on Sn-Bi over brass, suggesting that whiskers occurring under this plating condition do not necessarily depend on the type of environmental exposure. All MSA-based baths have produced either whiskers or nodules during the course of temperature-humidity exposure.

Table 2 Qualitative Results of Whisker Presence for All the Tests

Electroplating	Current density	Substrate	Pre-test	Indentation	Temperature Cycling	Temperature Humidity
Sn (1)	60 mA/cm ²	Cu	No	Yes	No	Yes - nodules only
	120 mA/cm ²		No	Yes	No	Yes - nodules only
SnCu	60 mA/cm ²	Cu	Yes - whiskers and nodules	Yes	Yes	Yes - whiskers and nodules
	120 mA/cm ²		Yes - whiskers and nodules	Yes	Yes	Yes - whiskers and nodules
SnCuPb	60 mA/cm ²	Cu	Yes - whiskers and nodules	Yes	Yes	Yes - whiskers and nodules
	120 mA/cm ²		Yes - whiskers and nodules	Yes	Yes	Yes - whiskers and nodules
Sn (2)	25 mA/cm ²	Cu	No	No	No	No
		Brass	No	No	No	Yes - whiskers and nodules
		Alloy 42	No	No	No	No
Sn-Bi	150 mA/cm ²	Cu	No	No	Yes - nodules only	No
		Brass	No	No	Yes	Yes - whiskers and nodules
		Alloy 42	No	One whisker	Yes	No

Table 3 Quantitative Whisker Results for Indentation and Temperature Cycling Tests

Electroplating	Current density	Substrate	Indentation		Temperature Cycling		
			Avg. # of Whiskers around Indentation	Longest Whisker (μm)	Whisker Density #/mm ²	Avg. Whisker Length (μm)	Longest Whisker (μm)
Sn (1)	60 mA/cm ²	Cu	8	6	0	0	0
	120 mA/cm ²		12	8	0	0	0
SnCu	60 mA/cm ²	Cu	20	10	15	6	15
	120 mA/cm ²		13	10	12	5	15
SnCuPb	60 mA/cm ²	Cu	15	9	39	6	6
	120 mA/cm ²		13	5	108	5	8
Sn (2)	25 mA/cm ²	Cu	0	0	0	0	0
		Brass	0	0	0	0	0
		Alloy 42	0	0	0	0	0
Sn-Bi	150 mA/cm ²	Cu	0	0	0	0	0
		Brass	0	0	196	7	14
		Alloy 42	1	2	4019	18	41

Discussion

For all Sn, Sn-Cu, and Sn-Cu-Pb over copper samples produced in the Supplier 1 bath, the indentation test produced whiskers. With the exception of the Sn-Bi-plated alloy 42 sample, no whisker growth was induced by indentation for the Supplier 2 and Supplier 3 plating baths. If we compare these tests based only on the presence of whiskers, we can make some intriguing observations. In 9 out of 12 (75%) instances, the presence of whisker growth between indentation tests to temperature cycling tests matched. The same level of correlation was observed between indentation-induced whiskers and temperature/humidity-induced whiskers. For temperature cycling and temperature/humidity, the presence of whiskers matched 8 out of 12 times. Combining the presence of growth either by temperature cycling or temperature humidity, we find an 83% (10 out of 12) correlation with indentation-induced whisker growth. The indentation test failed to identify Sn over brass and Sn-Bi over brass as whisker producers.

Measured whisker lengths were found to be below disqualification length of 50 microns for all tests and all supplier plating baths. With regard to whisker length, the indentation and temperature cycling tests both found the Supplier 1 (MSA-based baths) Sn-Cu specimens to have the longest whiskers. For the indentation test, a different load and dwell time may have produced different results. Moriuchi et al. [8] found length to increase with load level and

dwell time. This finding was also observed by Shibutani et al. [9] and Yang and Li [10].

Conclusions

An indentation test method for evaluating tin whisker growth propensity was presented. The indentation test was applied to 12 different types of samples: MSA-based Sn, Sn-0.7Cu, and Sn-0.7Cu-1.3Pb plated over Cu substrate at either 60 or 120mA/cm², sulfuric acid-based Sn-plating, and Sn-Bi plating over Cu, brass and alloy 42. Whisker growth induced by temperature cycling and temperature humidity was compared with whisker growth induced by the indentation test. The presence of indentation-induced whiskers matched the presence of combined temperature cycling and temperature/humidity 83% of the time. In no case did the observed whisker growth exceed 50 microns, which was set as a failure criterion by IEC60068-82-2. From current results, the indentation test may provide value in evaluating whisker growth propensity, but further study will be required.

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