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Examination of Nickel Underlayer as a Tin Whisker Mitigator

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Abstract

Tin (Sn) whiskers are electrically conductive crystal structures that may spontaneously erupt from Sn-finished surfaces. Tin whiskers present a reliability hazard in electronics, because of their potential to create unintended short circuits. In the past, researchers have suggested that the addition of a nickel (Ni) underlayer between the copper (Cu) base material and Sn plating may act as a mitigation strategy for whisker formation. To evaluate this claim, samples with Sn plated directly on Cu, and samples with a Ni underlayer between the Sn and Cu, were subjected to environmental exposure to induce whisker growth. Prior to the test, all samples were stored in an office environment for 2.5 years and little to no whisker growth was observed. Testing consisted of 1000 temperature cycles (-55°C to +85°C, 10 min dwells) followed by two months of elevated temperature humidity exposure (60°C and 85%RH). During the test, whisker length sand densities on the samples were periodically measured. All whisker lengths were measured in accordance with the JESD201 standard, taking the effective shorting distance of the whisker (between the root of the whisker and the point farthest away). We present a simple methodology for whisker length measurement as an alternative to the JEDEC-suggested technique of varying the observation angle of the whisker to see its maximum length.

All of the samples were found to have whiskers after the first 500 temperature cycles. Further exposure to temperature cycling and elevated temperature/humidity did not significantly increase whisker density. Samples with the Ni underlayer had a greater average whisker density (around 2900 whiskers/mm^2) compared with samples where the Sn was plated directly on the Cu (around 1800 whiskers/mm^2). During temperature cycling, whisker lengths were similar for both sets of samples, with average lengths of around 12µm. Elevated temperature humidity exposure induced whiskers with lengths greater than 200 µm exclusively on samples with a Ni underlayer.

Upon completion of the experiment, whisker length and diameter data was gathered from 877 whiskers. No correlation was found between whisker diameter and its length. In addition, whisker growth angles were calculated for 588 whiskers, and then binned in 10° intervals to see whether any preferential growth orientation existed. The results demonstrated the absence of favored growth angles; however, very few whiskers grew at angles close to the surface. Measurements of plating thickness using X-ray Fluorescence (XRF) revealed that two specimens had a Sn plating thickness of 4.5µm while the remainder had thicknesses ranging from 6.7µm – 9.5µm. A thickness of 1.2µm was measured for Ni on specimens with a Ni underlayer. Distinctly fewer whiskers were found on the 4.5µm Sn finish (less than 200 whiskers/mm2 compared to the 2000-4000 whiskers/mm2 seen on thicker-plated Sn). However, longer whiskers were found on the thinner plating. Observations 1 year after exposure to the environmental test conditions found no further changes in whisker lengths or densities. Thus, massive whisker growth appeared to be due solely to exposure to the environmental test conditions.

Introduction

The first report of tin whiskers dates back to an accidental finding in 1947 of β-tin filamentary protrusions out of polished tin-aluminum alloys [1]. Over the next 20 years, tin (Sn) whiskers were recognized as a threat to the electronics industry and were thoroughly studied by Bell Telephone Labs [2,3]. Despite this warning, electronic part and equipment manufacturers continue to suffer periodically from tin whisker formation. In research related to tin whiskers, small amounts of lead in the tin finish have been documented to significantly retard whisker formation, although not necessarily eliminate it [4]. However, a European-driven ban on the use of lead in electronic products has resulted in selection of pure tin and tin based lead-free finishes for electronic device terminations. The selection of pure tin and tin-based lead-free terminal finishes is intended to maintain compatibility with existing tin-lead assembly processes and lead-free solders which are also tin-based alloys.

To reduce the propensity of tin whisker formation, part manufacturers have applied various mitigation strategies in plating tin. One is the application of annealing, usually 150°C for 1 hour immediately after plating. The annealing step may reduce whisker growth propensity by reducing internal stress due to the plating process, increasing grain size, and promoting a uniform interfacial intermetallic boundary layer between the tin film and the substrate [5,6]. A alternative method is the use of a nickel underlayer [7,8]. In addition to a barrier layer and annealing, a minimum plating thickness of 8 microns has been suggested to reduce the impact of local stresses that arise from the formation of intemetallic compounds at the tin-substrate interface [9].

The industry has put forward several documents as guidelines in assessing tin whisker growth on tin-rich finishes, namely JESD22-A121A [10] and IEC 60068-2-82 [11]. These documents suggest environmental testing conditions for inducing whisker growth. Limited knowledge, however, exists with regard to comparing the whisker growth in these short-span stress tests to long-term ambient storage conditions.

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Whisker Growth Study

JESD22-A121A and IEC 60068-2-82 are intended to provide evaluation of newly plated specimens as a means of providing quality control of plating process. The tests outlined in these standards are conducted on independent sets of test specimens. It is unclear what impact extended storage and sequential application of test conditions would have on whisker formation. To this end, a test was conducted to (1) record time-sequenced whisker growth parameters; (2) evaluate the effectiveness of a nickel (Ni) barrier layer as mitigation against tin whiskers; and (3) compare long-term effects of whisker growth in environmental tests to ambient storage.

Test coupons were prepared with a copper (Olin 194 Cu-2.4Fe-0.03P-0.1Zn) substrate to simulate the substrate material commonly used in electronics industry. Individual coupons measured 31.7x12.7x0.5mm. A single commercial vendor electroplated all coupons with Sn with half of the specimens first plated with a Ni layer.

Surface Sn grain size averaged 4μm with a standard deviation of 1 μm. Using X-Ray Fluorescence (XRF), the thickness of tin plating was measured to average 7.5μm with a standard deviation of 1.7μm – further discussion is provided in Plating Thickness section below. On samples containing Ni, the underlayer thickness averaged 1.4 with a standard deviation of 0.2μm, which is close to the 1.27μm suggested minimal Ni barrier thickness [9].

After plating, samples were held in room ambient for 2.5 years. Over that period, no whisker growth was observed. Some samples were then put through sequential environmental testing, while others were left in ambient conditions as control (Table 1).

Table 1: Number of coupons in each category of the test

	Sn on Cu	Sn on Cu with Ni underlayer
Control (4 years of ambient exposure)	2	2
Test (sequential environmental exposure)	6	6

At the time of test initiation, only JESD22-A121 [12] (published May 2005) test conditions were available, and the test was conducted based on them:

- Temperature Cycling: -55°C to +85°C, 10min dwells, 3 cycles/hour
- Elevated Temperature Humidity: 60°C and 85%RH

Standards published later (including IEC 60068-2-82 and JESD22-A121A) have only changed the Elevated Temperature Humidity conditions to 55°C and 85%RH. Table 2 below shows the environmental exposure coupons went through during the test and the times that whisker growth parameters (length and density) were gathered. All whisker inspections were done using Scanning Electron Microscopy (SEM).

Table 2: Timeline of coupon exposure (with ● representing observations of whisker growth)

Control	4 years in ambient			
	●	● ●	●	●
Test	2.5 years ambient	1000 Temp Cycles	2 months Elevated Temp Humidity	1 year ambient

Gathering Length, Density, and Diameter Data

The length of a whisker was defined in accordance with JESD22-A121A with a single measurement of the effective shorting distance defining the whisker instead of the sum of lengths of the individual whisker segments. All of the current whisker testing standards suggest tilting a sample under SEM until the whisker is perpendicular to the field of view. Given the three-dimensional nature of whiskers, this technique is raises some concern, since viewing a whisker from an angle other than perpendicular to its length would give an underestimated value. Because whisker measurement by the above method is time-consuming and often impractical, we have chosen to use two views of a whisker taken from different viewing angles (10° in our case) and used the following formula to calculate true three-dimensional whisker length:

$$L_{ab} = \sqrt{\frac{L_{cd}^2 + L_{ce}^2 - 2L_{cd}L_{ce}\cos\theta}{\sin^2\theta} + \left(L_{cd}\tan\beta\right)^2}$$

Variables are identified below, and represented in Figure 1:

Axis along L_{ac} is the tilt axis

L_{cd} = projection of whisker length on axis perpendicular to tilt axis in Plane 1

L_{ce} = projection of whisker length on axis perpendicular to tilt axis in Plane 2

θ = tilt angle between Plane 1 and Plane 2

β = angle between L_{cd} and L_{ad} in Plane 1

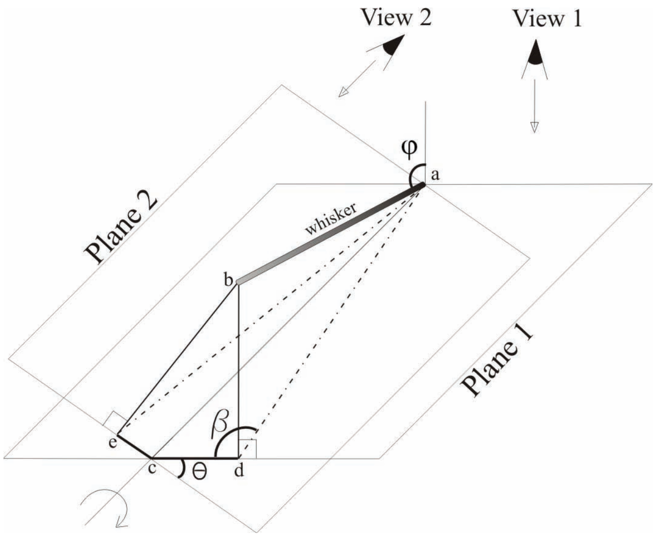

Figure 1: Diagram of whisker length measurement. All variables used for whisker length calculation are given above. φ is the growth angle of the whisker

Freeware Image J [15] was chosen to conduct length measurements from SEM images.

For the density measurements, areas of 260µm by 220µm were randomly chosen across each coupon, with 11 areas analyzed per coupon (66 per condition). For the purpose of comparison, some – but not all – areas and whiskers were returned to at various stages of the test to visually record the progression of growth.

Upon completion of the environmental exposure, after 1000 temperature cycles and 2 months in elevated temperature humidity, both length and diameters of whiskers were measured.

For a year following test completion, coupons were stored in ambient environment. After one year, previously inspected areas of each coupon were re-examined to update whisker length and density measurements. We shall note here that no changes were observed on the coupons between the end of environmental stress test and the completion of one year in ambient storage.

Length and Density Distributions

Prior to the test, about 2.5 years after plating, no whiskers were found. After the sequential environmental exposure, whisker density and length distributions were recorded and are documented in Table 3 and Table 4.

As previously mentioned, no additional whisker growth was observed one year of ambient storage following the end of the sequential environmental test. Control coupons that were not exposed to sequential environmental testing have remained whisker-free for 4 years of ambient exposure.

Table 3: Whisker density (# whiskers/mm^2) mean ± standard deviation at various stages of the environmental stress test. Each datum point represents 66 density measurements

	Sn on Cu	Sn on Cu with Ni underlayer
500 temp cycles	2707 ± 1320	1535 ± 1392
1000 temp cycles	3216 ± 955	1906 ± 1524
2 months in elevated temp humidity	2987 ± 999	1864 ± 1480

Whisker length data was gathered from measuring 300-600 whiskers at different observation intervals. Whiskers were chosen from the areas used for density measurement.

Table 4: Whisker length mean ± standard deviation at various stages of the environmental stress test

	Sn on Cu (µm)	Sn on Cu with Ni underlayer (µm)
500 temp cycles	9 ± 5	9 ± 5
1000 temp cycles	12 ± 5	12 ± 7
2 months in elevated temp humidity	12 ± 6	19 ± 18

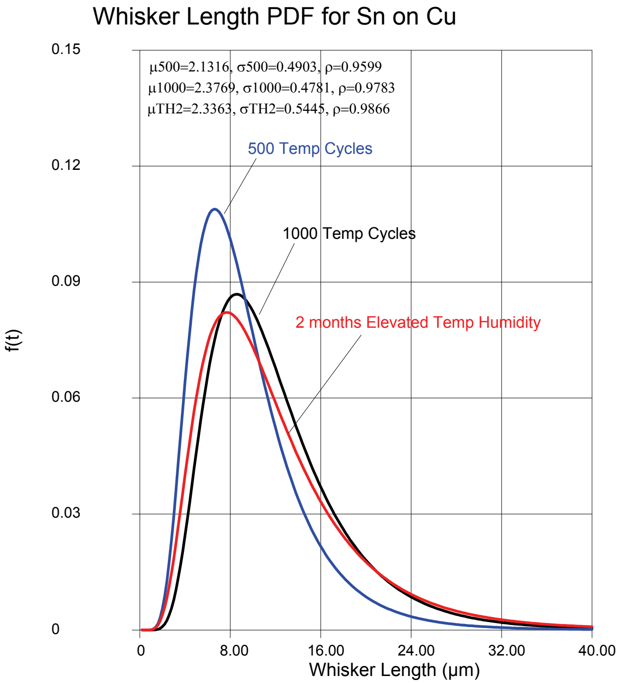

Figure 2: Whisker length distributions for Sn on Cu at three stages of the test

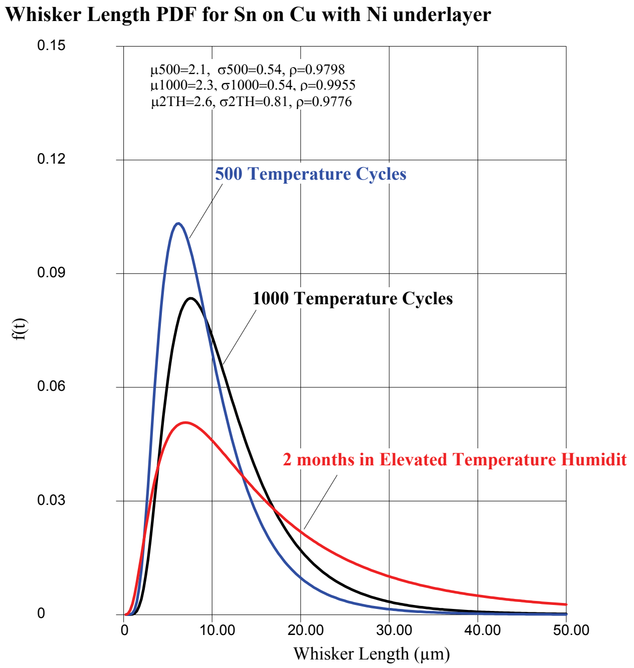

Figure 3: Whisker length distributions for Sn on Cu with Ni underlayer at three stages of the test

Consistent with observations made by Fukuda [13] and Fang [14], the length data closely followed a log-normal distribution, with parameters displayed in Figure 2 and Figure 3.

Data collected for both whisker density and length seemed to progress forward from 500 to 1000 temperature cycles, however, have off-set back for 2-months of elevated temperature humidity that followed. This was most likely due to measurement uncertainties, where new areas and new whiskers were include in density and length data sets. Note that variance has increased with each consecutive set of measurements.

Length and Diameter Measurements

After the sequential loading test, a total of 877 whiskers were randomly selected between the 12 samples for length and diameter measurements. Lengths were measured using JESD22-A121A standard, where a single shorting-distance length was calculated for each whisker.

Whisker lengths fell between 2μm and 256μm, while diameters ranged between 1μm and 14μm. Both the whisker lengths and whisker diameters fit lognormal distributions. The lognormal characteristic parameters are listed in Table 5, with histograms of whisker distributions given in Figure 4 and Figure 5.

Table 5: Lognormal Characteristic Parameters of Length and Diameter Distributions

	μ	σ	ρ
Length (μm)	2.59	0.70	0.9754
Diameter (μm)	1.48	0.40	0.9994

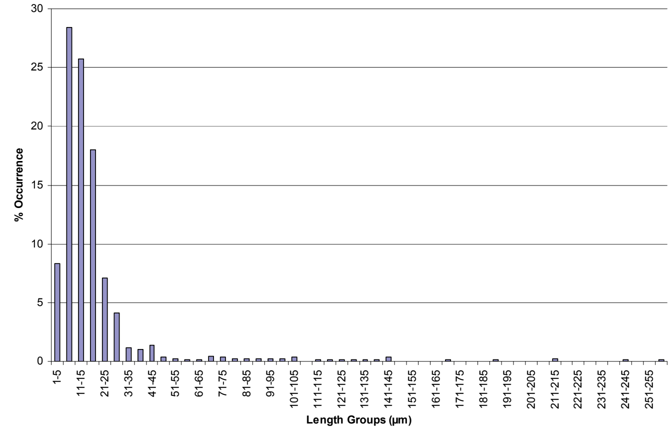

Figure 4: Histogram of 877 whisker lengths at the end of the test

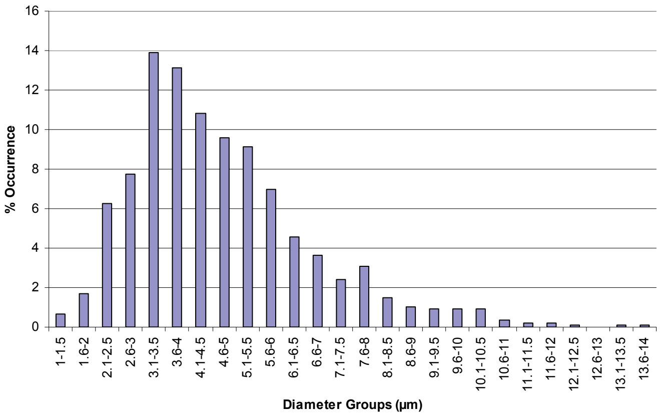

Figure 5: Histogram of 877 whisker diameters at the end of the test

Since formation of a whisker requires a large amount of atomic transport to the site of whisker growth, it was theorized that the total amount of tin delivered to the base of a whisker may be similar for all whisker. Thus, volume of whiskers would be constant, meaning that whiskers with smaller diameters would grow longer, while larger-diameter whiskers would be shorter. If the volume of a whisker is kept constant, and we assume that a whisker has a constant circular cross-section that can be measured as a diameter of a whisker, then a correlation would exist between whisker lengths and whisker diameters.

To test this theory of constant whisker volume, the 877 whisker were plotted on a diameter vs. length graph as shown in Figure 6. The resulting plot is a classic representation of non-correlation, as is confirmed by a correlation coefficient of -0.06.

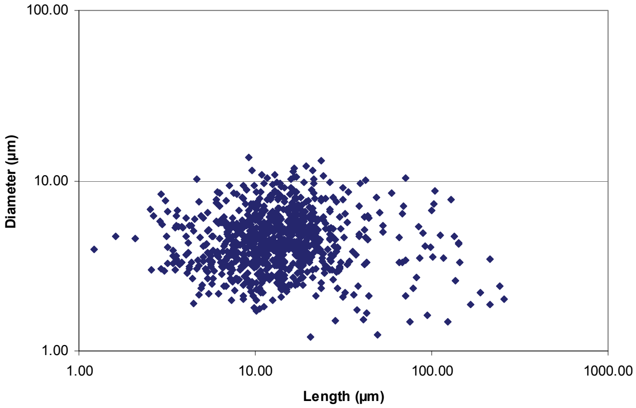

Figure 6: Diameter vs Length correlation for 877 whiskers measured at the end of sequential environmental exposure: correlation coefficient is -0.06

In the above plot, no differentiation was made between different samples, underlayer presence, or the size of the whiskers used in order to create the diameter-length correlation. To see whether some factors do play a role, the data was separated into groups to check if diameter-length correlation existed for some subset of the data. Several grouping attempts were made:

- Samples with Ni underlayer, apart from samples with no underlayer present;
- Each of the 12 samples separately;
- Only whiskers with ratio of length:diameter ratio greater than 5:1;
- 5:1 ratio in conjunction with underlayer separation;
- 5:1 ratio on each of 12 samples separately.

None of the above groupings yielded noticeable correlation.

However, whiskers were originally measured using the JESD22-A121A standard, where whisker length is reduced to a single line from the root of the whisker to its furthest point. Thus, no account was made for whiskers that had bends/kinks along their lengths. The total volume of a whisker with bends would be greater than the volume of its single-line simplification. Therefore, the above data is an underestimate of the total length of whiskers that would be used in volume calculation.

To re-test the hypothesis of whisker diameter to length correlation, some whiskers were chosen to be re-measured by JESD22-A121 method, where the individual segments of a whisker a measured and later on summed up to get the total length. For practical considerations, not all whiskers were looked at, only ones that had length : diameter ratios of greater than 5:1 upon the first measurement (where length was equal to the shorting distance) were selected from the above data and re-measured. The results were still indicating no correlation present between the diameter of the whisker and its length.

Whisker Growth Parameters as a Function of Plating Thickness

It was noted previously that plating thickness of all samples was measured using X-Ray Fluorescence (XRF). Average thickness of tin across all 12 samples was 7.5µm with standard deviation of 1.7µm. The spread of values is indicative of the variations within a commercial plating process – the nominal plating thickness for the parts may not always be representative of the true values. A summary of plating thickness and whisker growth metrics is presented in Table 6.

Table 6: Plating thicknesses along with average length and density values for each sample at the completion of test

Sample#	Sample Description	Ni underlayer (µm)	Sn plating (µm)	Average Length (µm)	Max Length (µm)	Average Density (#/mm^2)
1	Sn on Cu, Ni underlayer	1.6	9.5	13	66	3573
2	Sn on Cu, Ni underlayer	1.6	8.5	14	50	1493
3	Sn on Cu, Ni underlayer	1.6	8.9	20	244	3337
4	Sn on Cu, Ni underlayer	1.3	4.5	30	214	126
5	Sn on Cu, Ni underlayer	1.3	4.5	30	256	185
6	Sn on Cu, Ni underlayer	1.3	9.1	22	213	2531
7	Sn on Cu		8.6	10	20	2556
8	Sn on Cu		6.8	14	39	2793
9	Sn on Cu		8.7	10	21	2192
10	Sn on Cu		7.2	12	27	3317
11	Sn on Cu		6.7	13	32	2984
12	Sn on Cu		7.5	12	24	3956

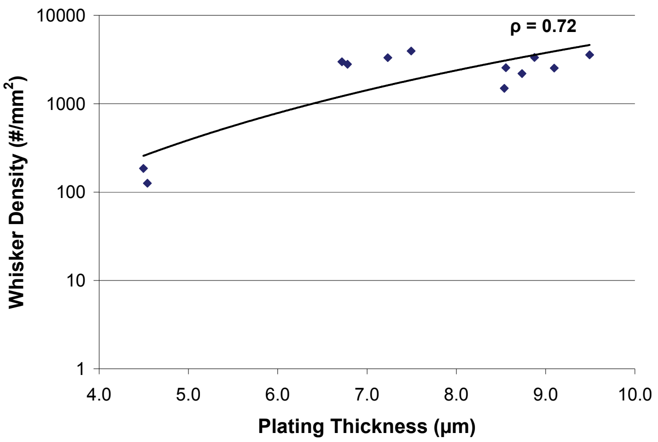

Figure 7: Correlation between whisker density and Sn plating thickness

Plating thickness appears to be related to the whisker density and length as can be seen in Figure 7 and Figure 8. Thicker plating does seem to induce more whisker growth, while average whisker length is greater for thinner coatings. Both whisker densities and lengths appear to be equally distributed along the higher plating thickness values (7-9µm), while a distinct difference exists at lower thickness (4.5µm). Maximum whisker lengths observed could be correlated to plating thickness: whiskers in 200-300µm range existed on both thicker and thinner plating.

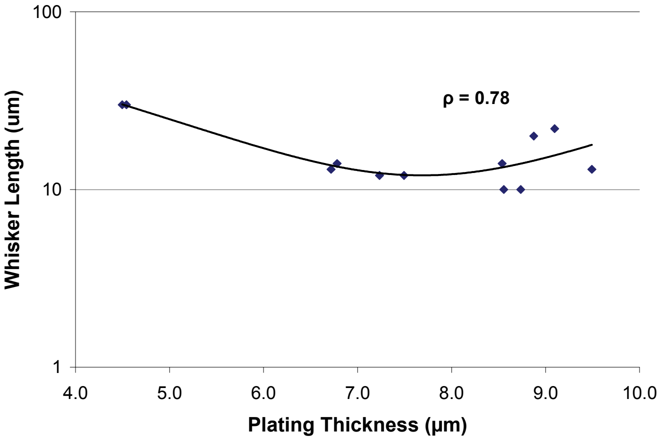

Figure 8: Correlation between whisker length and Sn plating thickness

Whisker Growth Angle

As part of the study, 588 whiskers were selected for growth angle estimation. The number of whiskers used in the growth angle estimation is less than for length and diameter distribution presented above. Our decision to ignore some whiskers was based on their shape – whiskers that generally were shorter than 10µm and at the same time ended up curling into an arc were ignored for angle calculations, due to the difficulty of assigning the growth angle for them. The growth angle in this case was taken to be between the effective shorting length line and an axis orthogonal to the surface, meaning that a whisker was first fitted with a single line to represent its length. The distribution of growth angles is given **Figure 9** with very few whiskers growing parallel to the surface in the 81°-90° range.

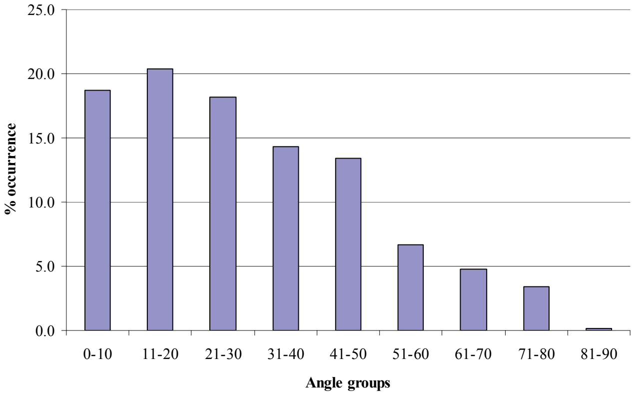

Figure 9: Growth angle distribution for 588 whiskers. Growth angle defined between the whisker effective shorting length and the axis perpendicular to the surface

These findings were consistent with previously reported observations of whiskers not having a preferential angle of growth and being less prone to grow parallel to the surface [16,17]. (Note: Hilty [16] defined the growth angle between the surface orthogonal and the whisker, while Fang [17] measured the angle from the surface to the whisker).

Conclusions

A whisker study compared Sn plated over Cu substrates and Sn plated over Cu with Ni underlayer sandwiched in-between. All test samples have been stored in ambient for 2.5 years, then subjected to 1000 temperature cycles, followed by 2 months of elevated temperature humidity exposure. No growth was observed prior to the start of the test. During temperature cycling, a large number of whiskers grew on all coupons; however, their lengths were not exceeding 50µm. Elevated temperature humidity exposure added few whiskers, but the new whiskers were longer, sometimes exceeding 200µm in length. One year of ambient exposure after the test completion has not affected any whisker growth – no new whiskers were identified. Thus, it must be concluded that all whiskers were induced by the harsh environmental conditions, and their effects not carried over to the long-term ambient exposure of the coupons. Control coupons from the same plating batch that were stored in ambient for 4 years have shown no growth. These results raise questions as to the validity of environmental tests as representative of long-term whisker growth conditions.

The Ni barrier layer did not have any noticeable effect in suppressing whisker growth during the sequential environmental test exposure. On the contrary, coupons with Ni underlayer produced the longest whiskers under elevated temperature humidity conditions.

Quantitative data on whisker length, density, and diameter distributions were collected. Whisker lengths and diameters fit log-normal distributions. No correlation exists between whisker diameter and length, meaning that whiskers of varying thicknesses have equal chances of growing long or staying short. Growth angles of whiskers were measured, and it was shown that no preferential orientation exists, although whiskers tend not to grow close to the surface.

Some correlation between the thickness of Sn plating and the whisker density was observed. Independent of Ni underlayer presence, thinner Sn plating produced fewer whiskers. Average whisker length was also higher for coupons with thinner Sn plating, although maximum whisker lengths could not be correlated to plating thickness.

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