



Evaluation of Environmental Tests for Tin Whisker Assessment

Masters of Science Thesis Defense

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Committee:

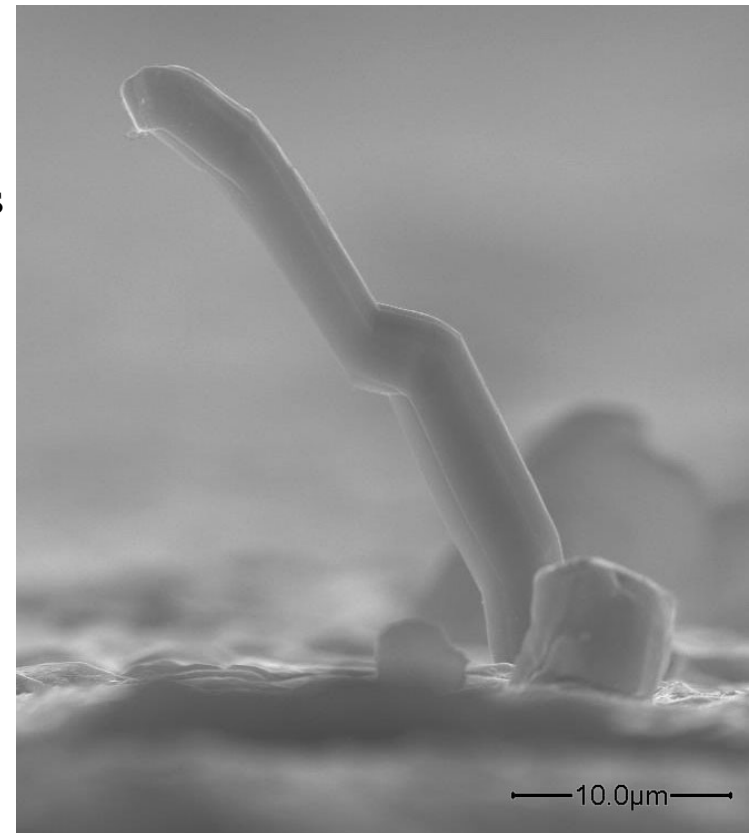
Dr. Michael Osterman, Chair

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Prof. Abhijit Dasgupta

Tin Whisker: an Introduction

- Tin Whisker - electrically conductive crystalline structure of tin that over time may grow outward from tin-rich surfaces
- Whiskers are formed through addition of atoms at the base, not the tip
 - Lengths vary from few micrometers to millimeters
 - Thicknesses range typically 0.5-10 μ m
 - Whisker densities may range from just a few whiskers to thousands per component
 - Whisker Growth may take hours, days, or years
- Long range diffusion responsible for tin transfer to site of whisker growth
- Types of Failures induced by Whiskers:
 - Electrical short circuit
 - ***Permanent*** if Current < Melting Current
 - ***Intermittent*** if Current > Melting Current
 - Metal Vapor Arc
 - Applications with high levels of current and voltage may cause whisker vaporizing into conductive plasma of metal ions
 - Plasma forms an arc capable of sustaining hundreds of amps
- Lead (Pb) has been demonstrated as whisker mitigator



Problem Statement

- Lack of understanding in error produced by existing whisker length measurement technique
- Lack of data relating environmental stress tests to tin whisker growth
 - Need to compare growth in short environmental tests to long-term exposure
- Lack of quantified data to be used in reliability assessments of tin finishes
 - Whisker density (#/area)
 - Whisker lengths
 - Whisker growth angles
 - Whisker thicknesses

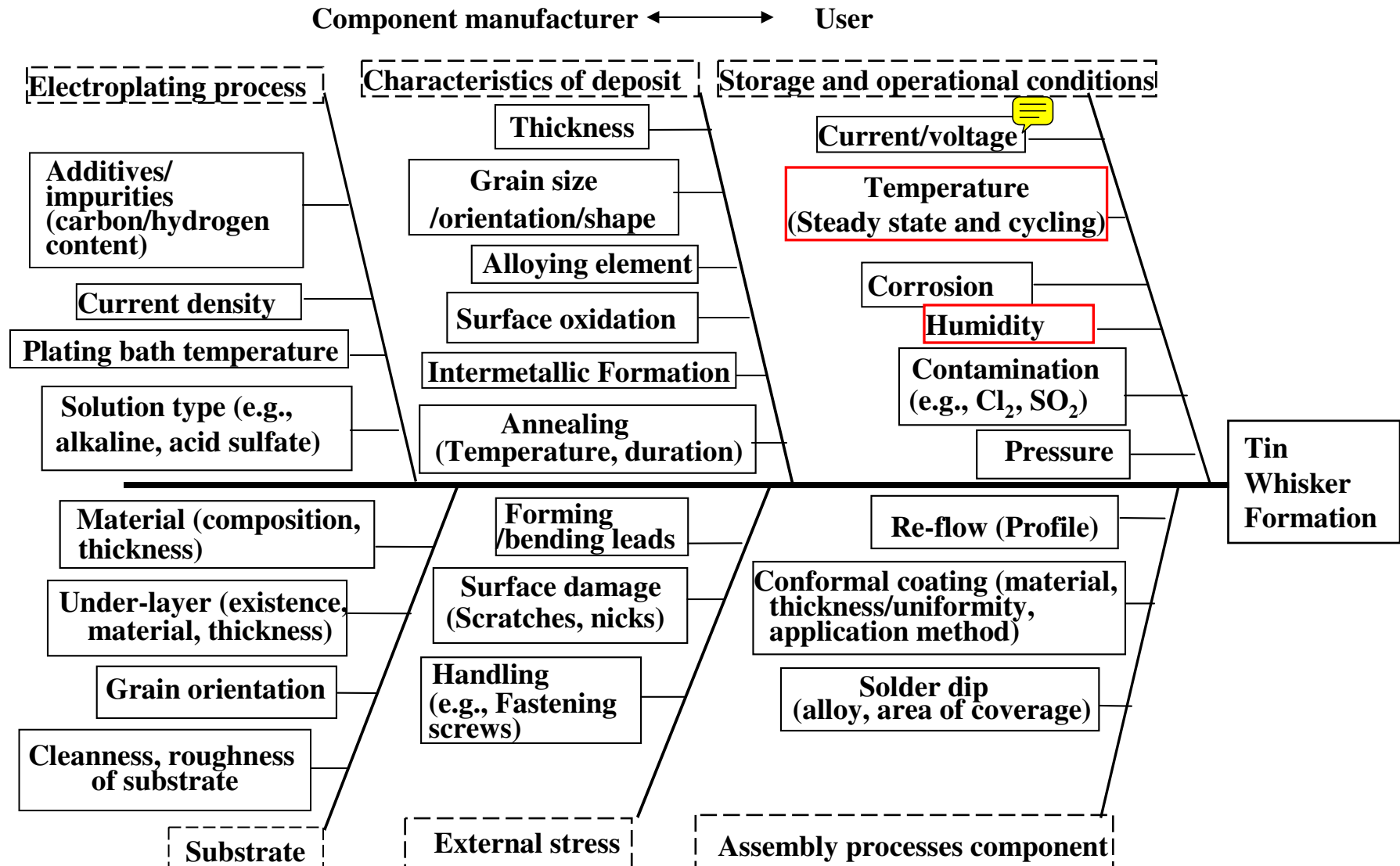
Standards for Assessing Whisker Growth

Standard	IEC60068-82-2	JESD22-A121A (†)	ET-7410
Issue Date	2007/5	2008/7	2005/12
Preconditioning	Soldering simulation Lead Forming	Reflow Lead Forming	Lead Forming
Ambient Storage	30°C, 60%RH 25°C, 55%RH 4000 hrs	30°C, 60%RH	30°C, 60%RH 4000 hrs
Elevated Temperature Humidity Storage	55°C, 85%RH 2000 hrs	55°C, 85%RH 60°C, 87%RH (*)	55°C, 85%RH 2000 hrs
Temperature Cycling	Min: -55°C or -40°C Max: 85°C or 125°C 1000 or 2000 Cycles	Min: -55°C or -40°C Max: 85 (+10/-0) °C 1000 or 2000 Cycles	-40°C to 85°C 1000 cycles
Acceptance Criteria	50µm	--	--

(†) JESD22-A121A does not prescribe duration of tests or Acceptance criteria. JESD201 should be used for that

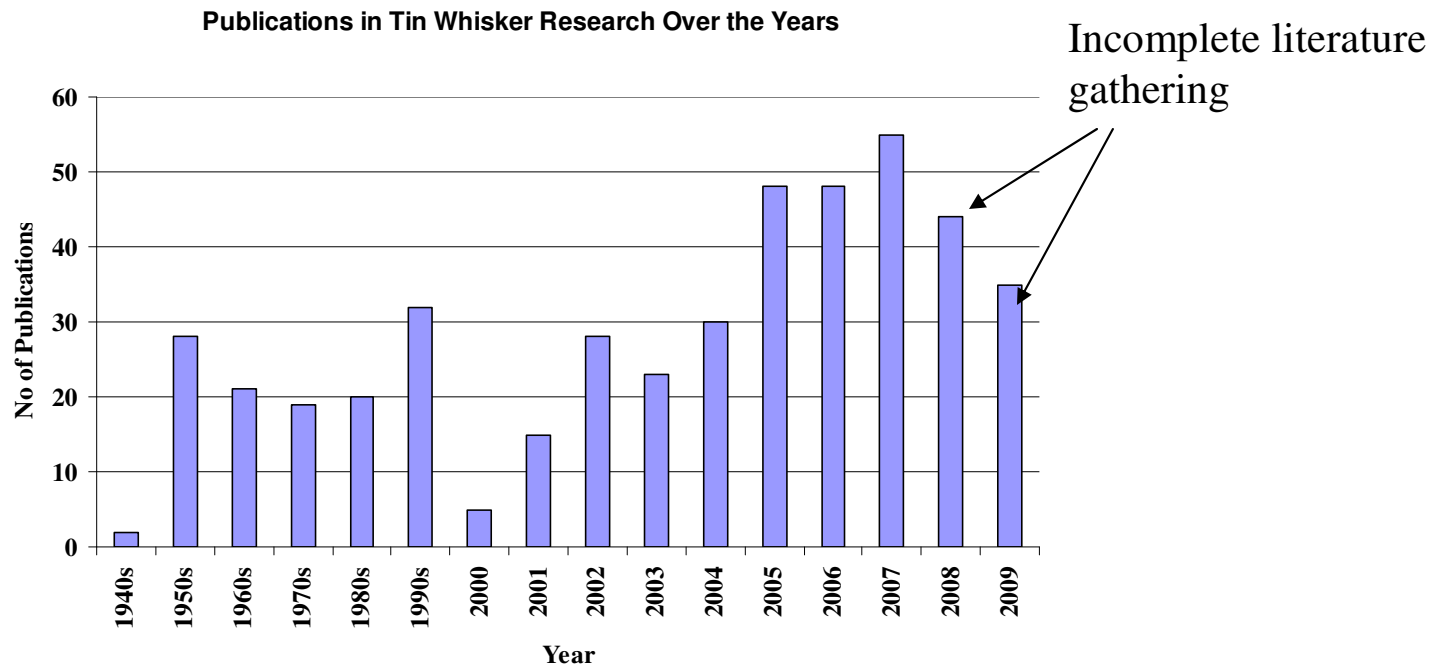
(*) Earlier version JESD22-A121, published May 2005

Factors Affecting Whisker Growth



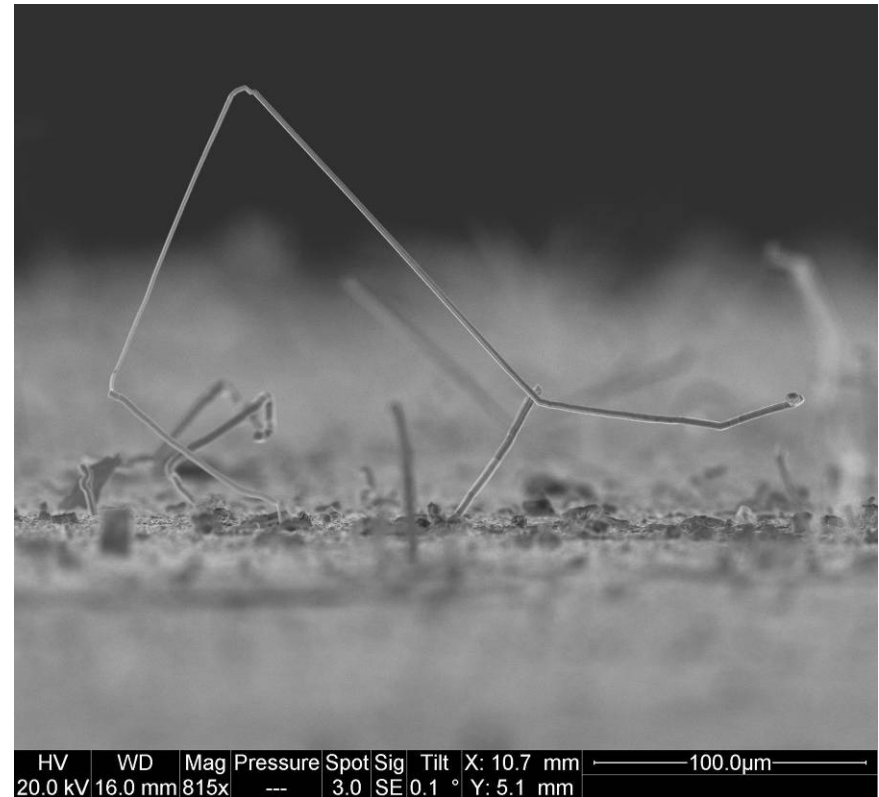
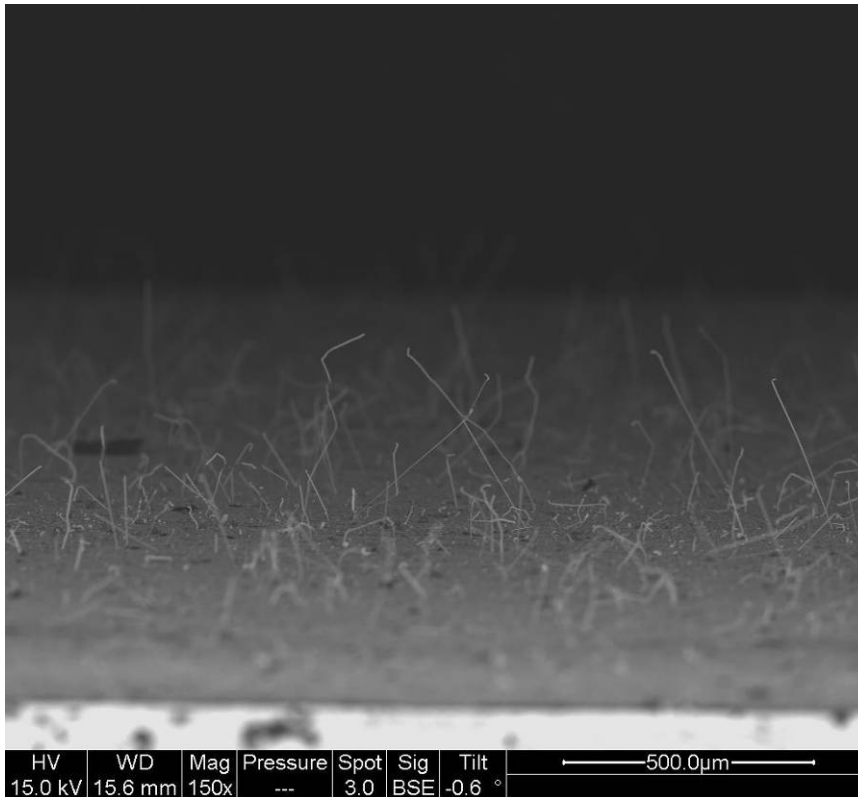
Brief Whisker Literature Summary

- ~450 papers addressing parasitic tin whisker growth published over a period of 63 years that whisker problem has been known
- Of them, 25% utilize temperature cycling and/or elevated temperature humidity tests
- None address long-term comparisons between environmental tests and ambient conditions



Practicality Issue in Measurements

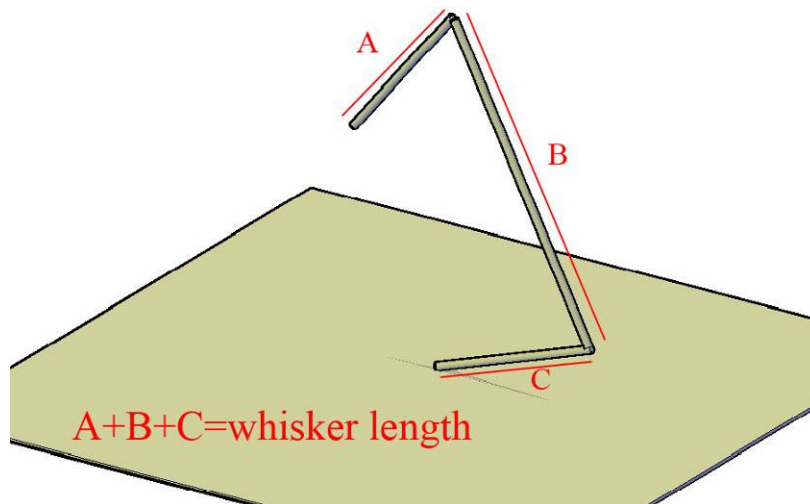
- Some whiskers exhibit complicated geometries
- Geometry of sample may not allow much degree of freedom, if need to rotate the specimens for better view
- Nevertheless, any modeling of whisker length requires a statistically significant number of whiskers to be measured



Whisker Length Definition

JESD22-A121(May 2005)

The distance between the finish surface and the tip of the whisker that would exist if the whisker were straight and perpendicular to the surface

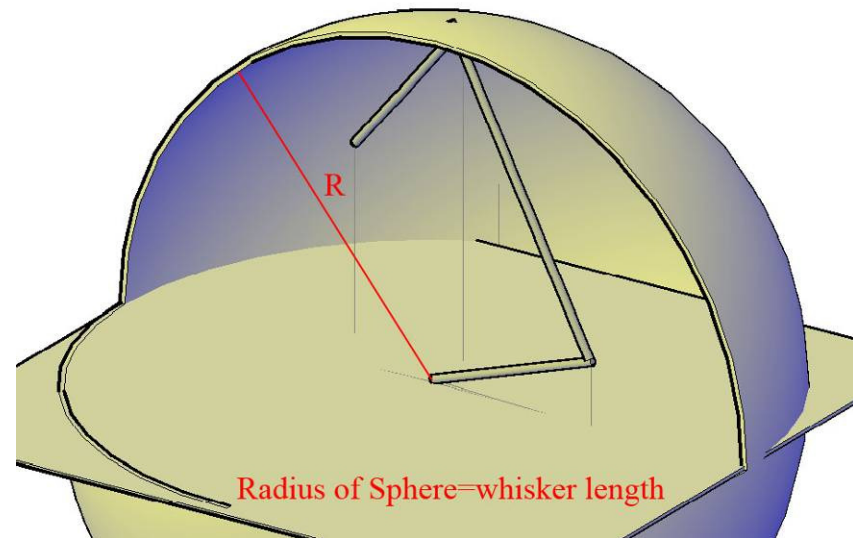


JESD201 (March 2006)

JESD22-A121A (July 2008)

IEC 60068-2-82 (May 2007)

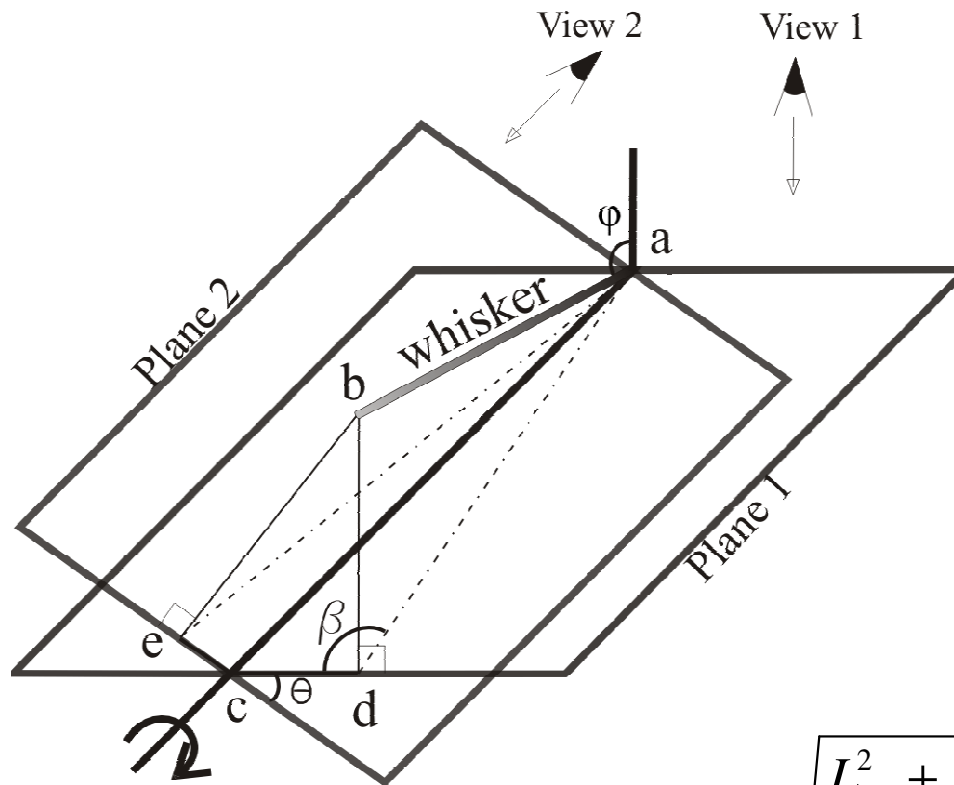
The straight line distance from the point of emergence of the whisker to the most distant point on the whisker



Guidance provided by JESD22-A121 in measurement technique: “... the system must have a stage that is able to move in three dimensions and rotate, such that whisker can be positioned perpendicular to the viewing direction for measurement”

Recommended Length Measurement

A more accurate measurement can be made by using two images offset by a known tilt



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

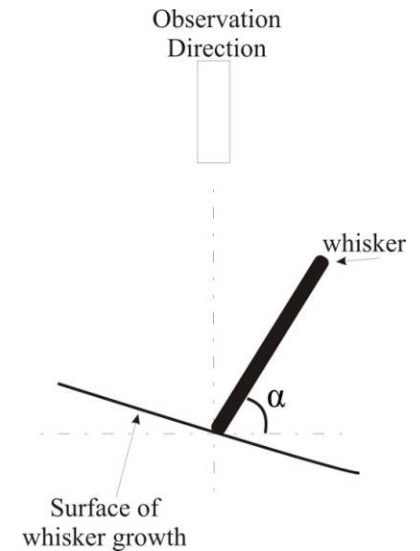
θ = tilt angle between Plane 1 and Plane 2

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

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

Whisker Length Measurement Errors

- If measuring whisker length from a single image:
$$\% \text{ Error} = (1 - \cos \alpha) * 100\%$$
- Measuring whisker lengths by tilting and aligning with field of view in SEM:
 - 7 participants
 - 3 whiskers
 - 20% \pm 11% error
 - 1.5 – 3hrs used up to measure 3 whiskers
- Measuring whisker lengths by two-images method:
 - 15 participants
 - 15 whiskers
 - 7% \pm 3% error
 - Images capture and measurements in under 10min



Evaluation of Environmental Tests

Questions:

- Do the environmental tests predict what whisker growth would exist, if this tin plating was stored in ambient conditions for several years?
- If comparing between whisker growth during environmental test and whisker growth during the same amount of time spent in ambient, is it correct to expect
 - either no growth anywhere (equivalent to non-whiskering tin)
 - or a far more prominent growth during environmental exposure as compared to ambient storage

Experimental Description


	Experiment 1	Experiment 2	Experiment 3
Plating	Commercial Sn. Half specimens with Ni underlayer	Commercial Sn electrolytes, plated in lab	Experimental Sn electrolytes, plated in lab
Substrate	Cu (Olin-194, Cu- 2.4Fe-0.03P- 0.1Zn)	Cu (C11000, 99.99% Cu)	Brass 260 (Cu- 30Zn)
Environmental Exposure	2.5yrs in ambient, + 1000 TC +2 months of ETH, + 2 yrs in ambient	1000 TC 3000hrs of ETH	1000 TC + 2 yrs of ambient 12 months of ETH + 1 yr of ambient
Control Exposure	5 yrs in ambient	150 days in ambient	

TC: Temperature Cycling

ETH: Elevated Temperature Humidity

Experiment 1: Results

average \pm STD



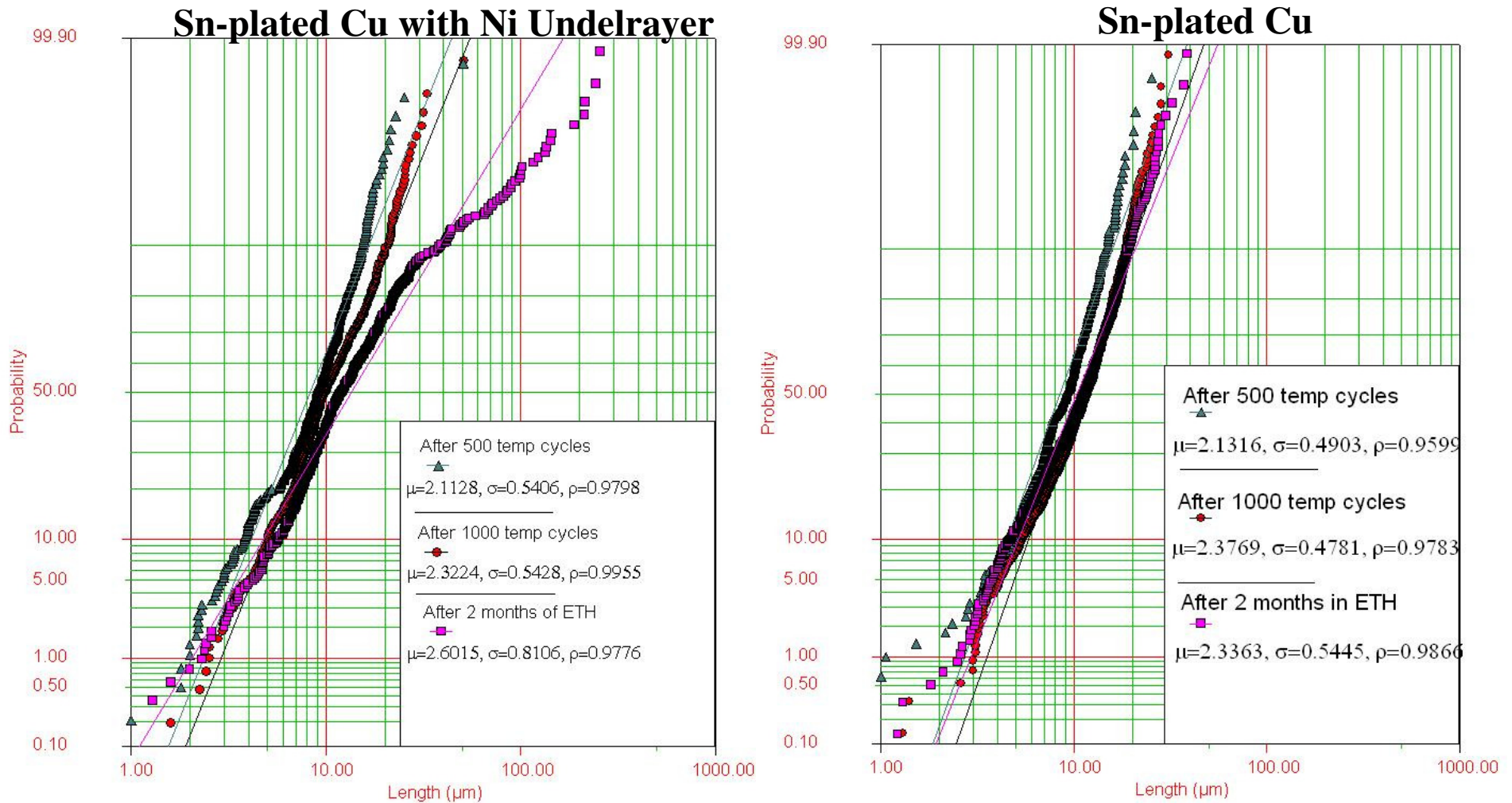
Storage Condition	Measured Parameters	Ni underlayer	No Ni underlayer
2.5 years in Ambient	No whiskers		
Temp Cycling (TC) 1000 cycles	Density (#/mm ²)	1907 \pm 1524	3216 \pm 955
	Avg Length (μ m)	12 \pm 7	12 \pm 6
	Max Length (μ m)	51	31
Elevated Temp Humidity (ETH) 60°C/87%RH 1500 hrs	Density (#/mm ²)	1864 \pm 1481	2987 \pm 1000
	Avg Length (μ m)	19 \pm 18	12 \pm 7
	Max Length (μ m)	256	39
Additional 2 years in Ambient	No change since Elevated Temp Humidity		

Ambient-stored control samples grew no whiskers during the 5-year test time

Experiment 1: Results in Graphs

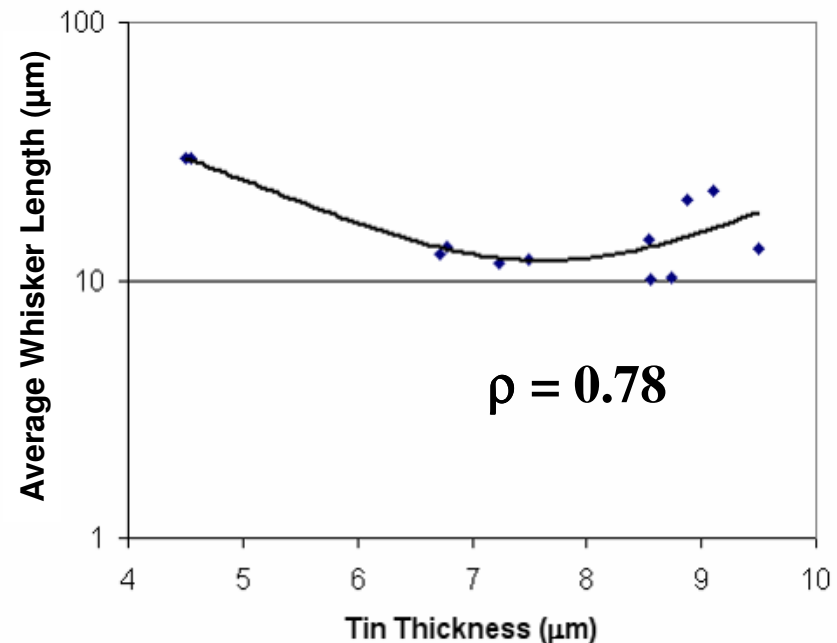
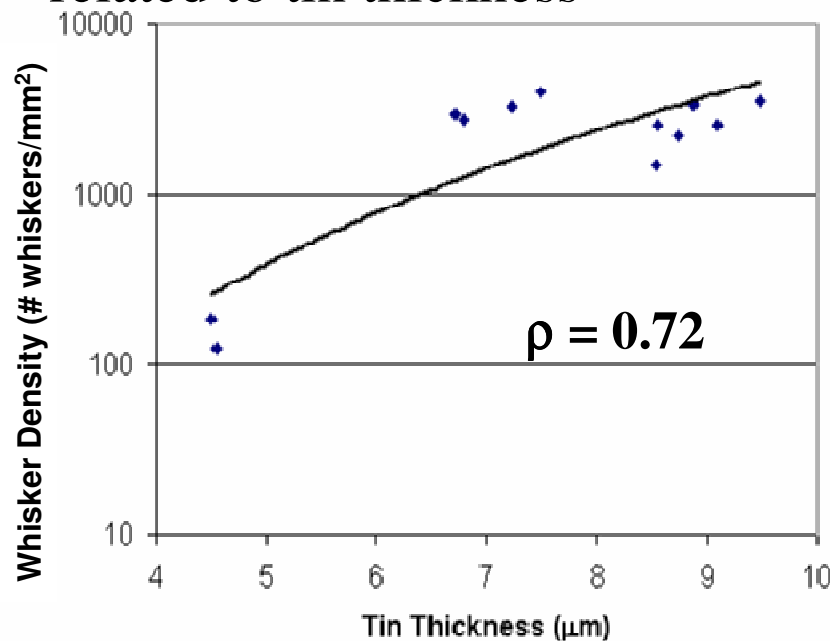
Lognormal cumulative probability distribution plot for whisker lengths at different exposure times

Note: Control ambient-stored specimens produced no growth



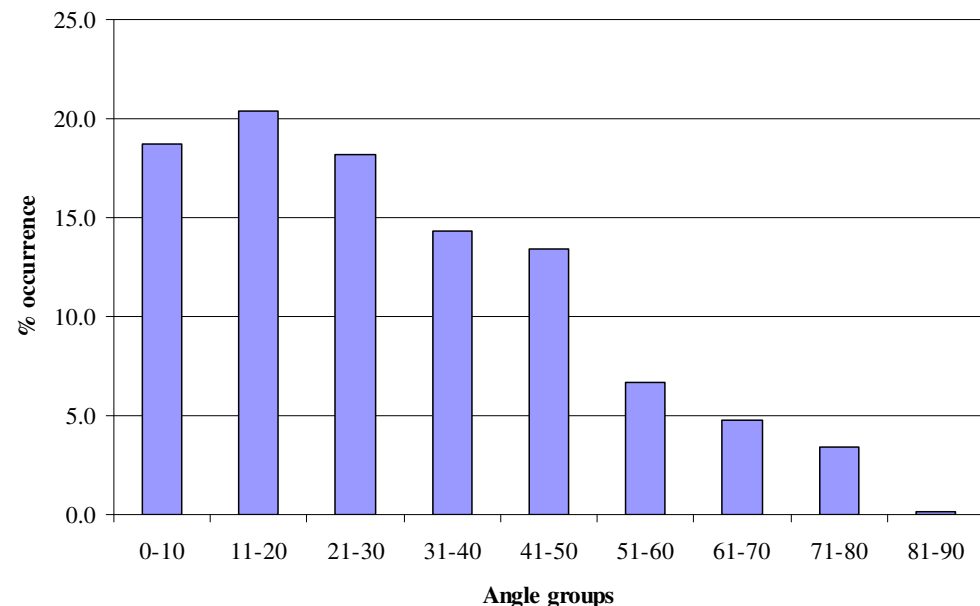
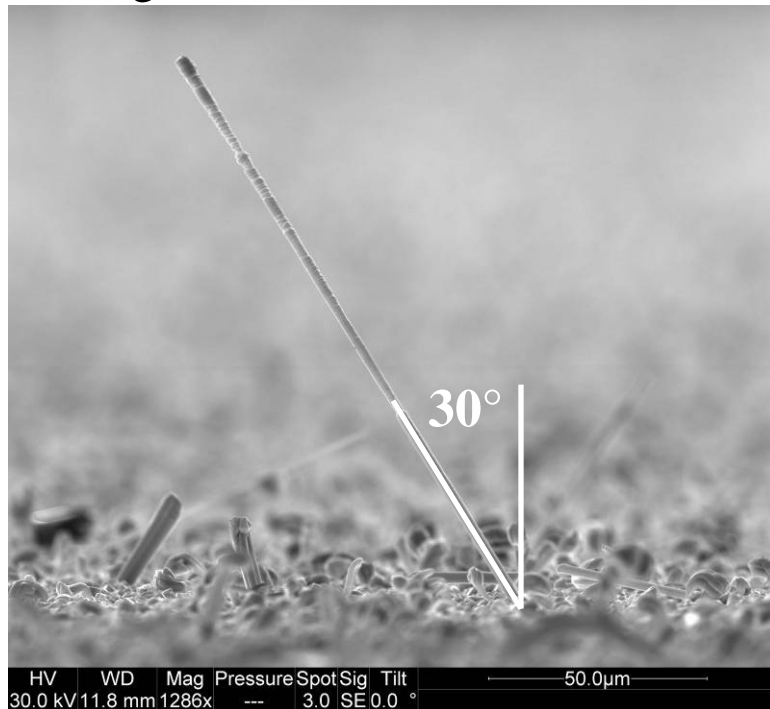
Experiment 1: Whisker Density and Length vs Plating Thickness

- Thickness measured using X-Ray Fluorescence (XRF)
- For the 12 samples (6 with Ni, 6 without Ni underlayer) used in environmental testing, tin plating thickness varied from 4.5 to 9.5 μm
- Ni underlayer thickness ranged from 1.2 to 1.5 μm
- Analysis of data indicates that both whisker density and lengths are related to tin thickness



Experiment 1: Whisker Growth Angle

- Growth angle measured between whisker and axis normal to surface
- No preferential growth angle seems to exist, but whiskers are less prone to grow close to the surface – same shown by Hilty[1] and Fang[2]



Note: Although not explicitly evident from this work, whisker growth angle CAN change during its growth period
See <http://www.calce.umd.edu/tin-whiskers/whiskermovies.htm> for examples

[1] R.D. Hilty, N. Corman, "Tin Whisker Reliability Assessment by Monte Carlo Simulation", IPC/JEDEC Lead Free Symposium, San Jose, CA, April 2005

[2] T. Fang, M. Osterman, S. Mathew, M. Pecht, "Tin Whisker Risk Assessment", Circuit World, Vol. 32 No 3, pp. 25-29, 2006

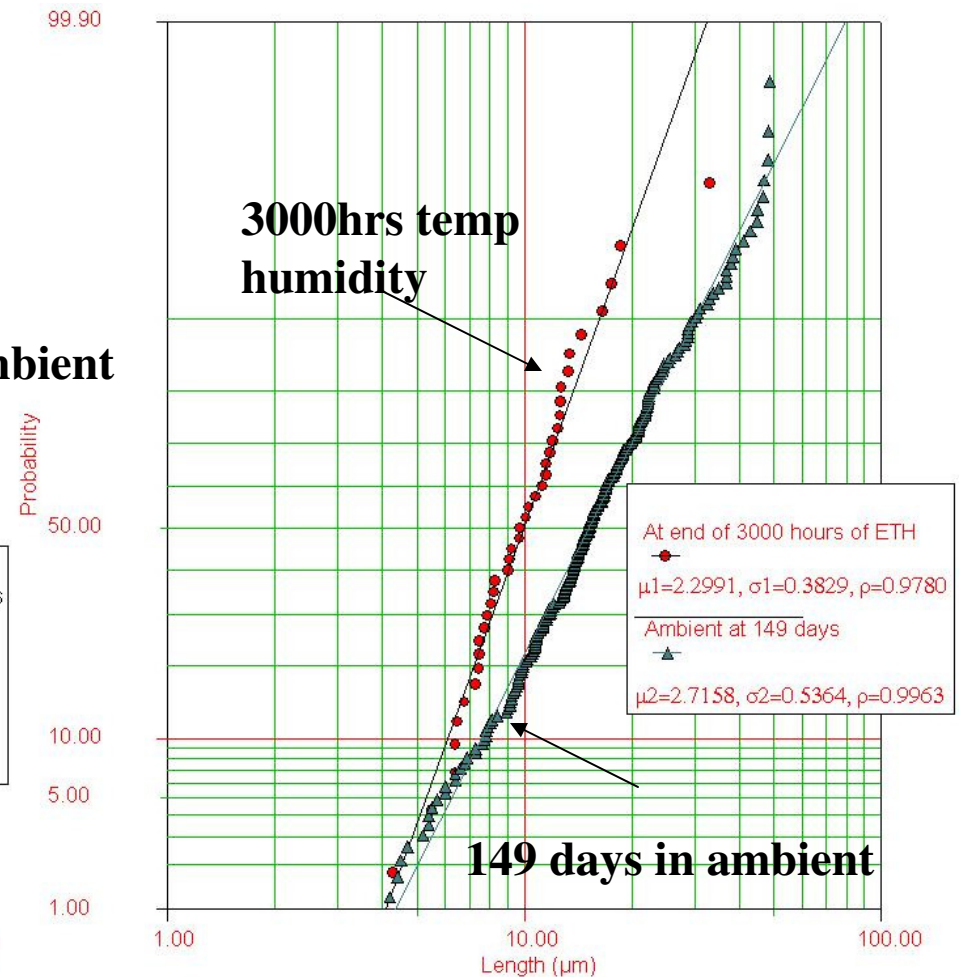
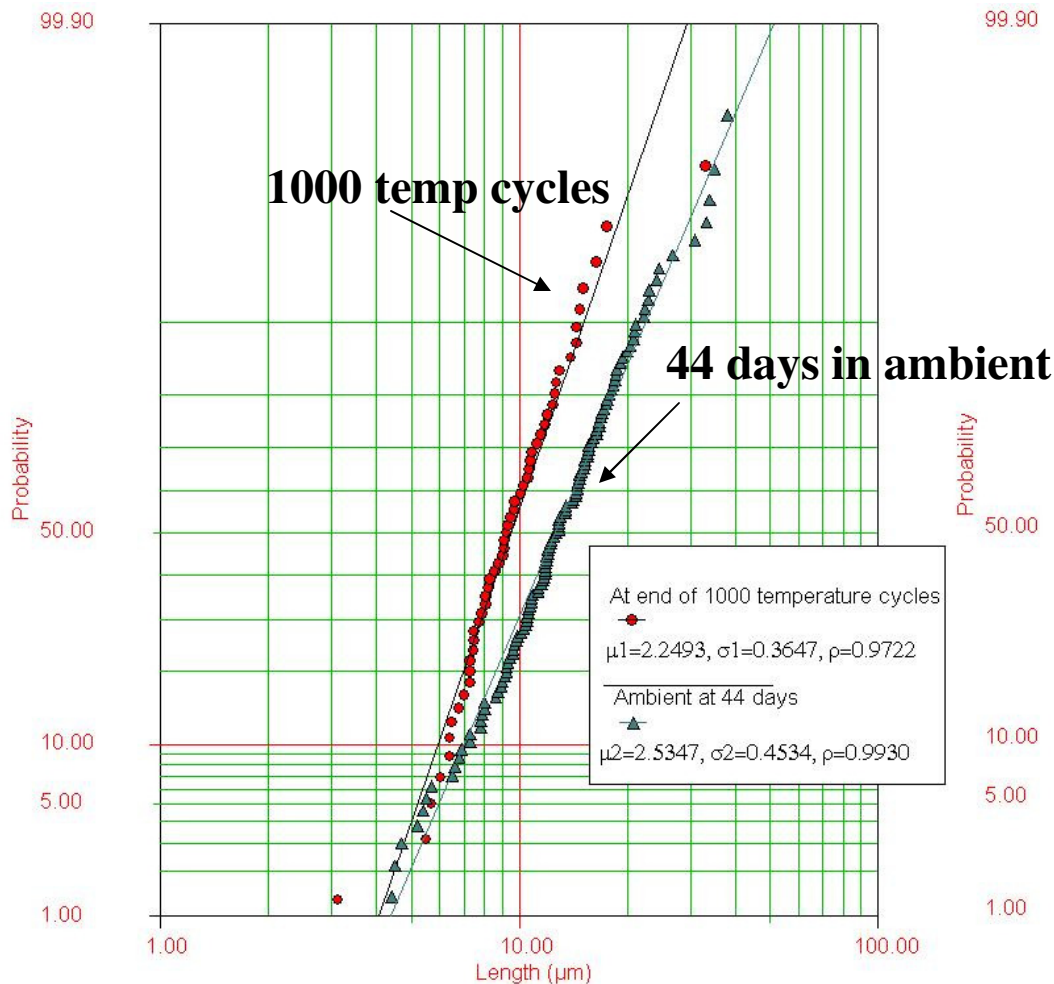
Experiment 2: Results

Exposure	Mean Density ± STD (whisker/mm2)	Mean Length ± STD (μm)	Maximum Observed Length (μm)
Temp Cycling (1000 cycles)	12 ± 9	10 ± 4	33
Elevated Temp Humidity (3000 hrs)	19 ± 1	11 ± 4	34
Ambient at 44 days (same as end of TC)	32 ± 16	14 ± 7	38
Ambient at 150 days (same as end of ETH)	41 ± 16	17 ± 10	49

Whisker growth in ambient exceeding (although not by far) that produced in any of environmental storage conditions

Experiment 2: Results in Graphs

Lognormal cumulative probability distribution plot for whisker lengths after end of exposure and corresponding control in ambient



Experiment 3: Results

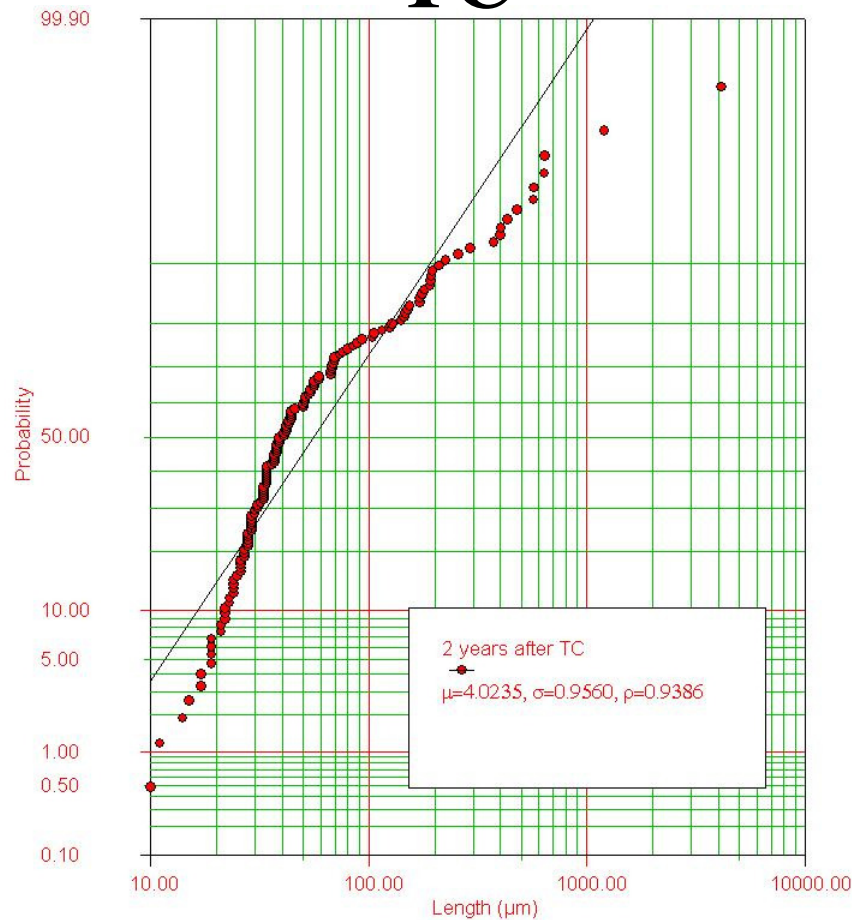
average \pm STD

Exposure	Inspection Interval	Whisker density (#/mm ²)	Whisker length (μ m)	Maximum whisker length observed (μ m)
TC	Pre-test	No whiskers		
	500 cycles	No whiskers		
	1000 cycles	No whiskers		
	1 year after TC	No whiskers		
	2 years after TC	24 \pm 12	125 \pm 181	4143
ETH	Pre-test	No whiskers		
	5 months	246 \pm 41	18 \pm 18	161
	9 months	285 \pm 135	31 \pm 28	194
	12 months	281 \pm 147	31 \pm 26	194
	1 year after ETH	281 \pm 147	31 \pm 26	194

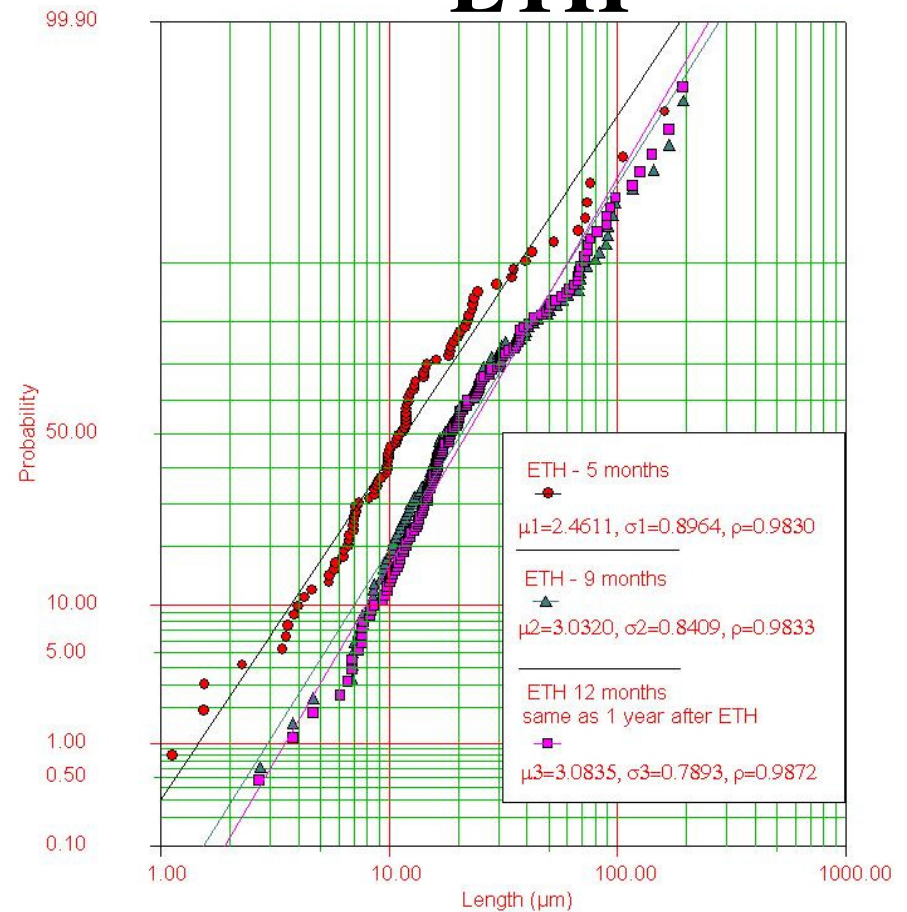
Experiment 3: Length Results

Whisker lengths fit to Lognormal distribution

TC



ETH



Summary of Environmental Exposure Results

Do the environmental tests predict the ambient-storage growth?

No. As currently implemented, they cannot be called predictive

As compared to ambient storage, whisker growth during environmental tests:

- Over-predicted the growth (experiment 1)
 - Whisker growth during environmental storage while no growth occurred before or after. Also, Ni underlayer was ineffective in preventing whisker growth
 - Control ambient specimens saw no whisker growth throughout 5-year period
- Had little distinction (experiment 2)
 - End of temperature cycling and elevated temperature humidity tests show almost the same (but slightly less) whisker growth as compared to ambient storage of same duration
 - Expecting whiskers on ambient-stored specimens to continue grow over time
- Under-predicted the growth (experiment 3)
 - No growth during temperature cycling or for 1 year following
 - Abundant growth after 2 years post-cycling (used as ambient control)
 - Whiskers grew during elevated temperature humidity exposure, but no growth seen throughout 1 year of post-exposure ambient storage, but severely shorter in length than that seen on ambient

Sequential Environmental Exposure

Questions:

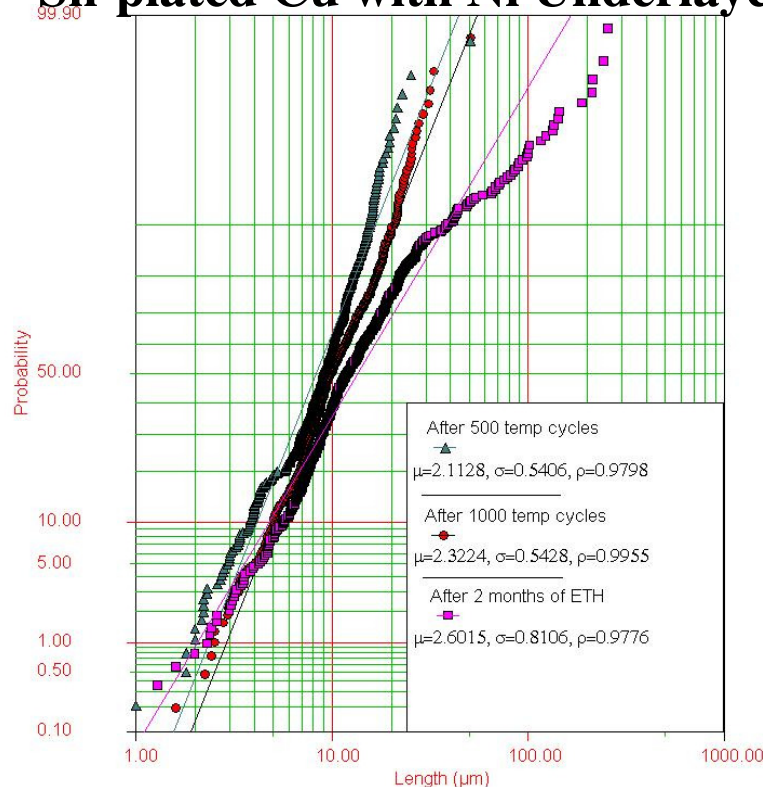
Does sequencing different environmental exposures produce more prominent whisker results, than any of the exposures separately?

Would it be preferred to utilize sequences of environmental exposures to assess a Sn plating for whisker growth?

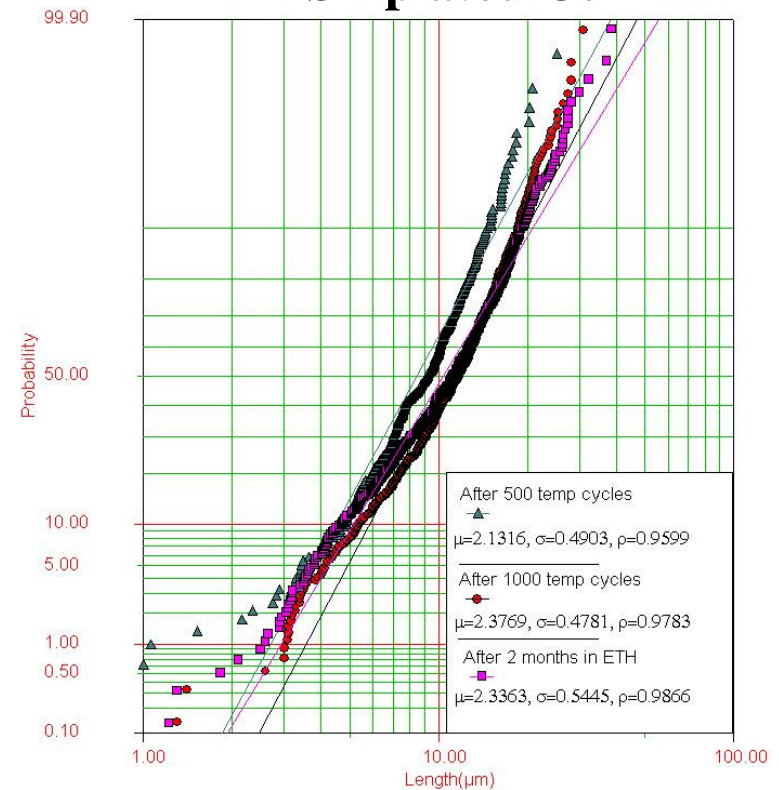
Experiment 1 Summary

- Experiment 1 has demonstrated that sequencing temperature cycling and elevated temperature humidity may result in longer whisker growth, although without much addition to whisker density

Sn-plated Cu with Ni Underlayer

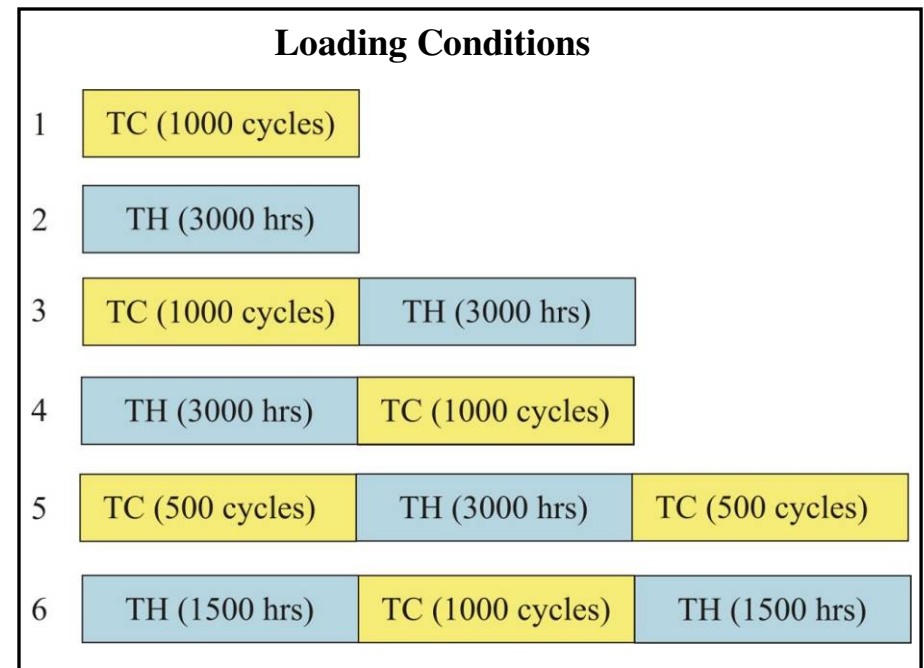


Sn-plated Cu



Experiment 2: Multiple Sequences of Environmental Exposures

- A total of 6 tests designed
 - Each is a combination of JEDEC
 - TC: Temperature Cycling -55°C/+85C, 10 min dwells.
Inspections conducted every 500 cycles
 - TH: Temperature Humidity 55°C/85%RH.
Inspections conducted every 1000 hours
- 21 samples total
 - 3 Samples for each of 6 conditions
 - 3 Control samples to be left in ambient
- Sample description:
 - 25mm x 25mm x 0.8mm
 - Cu substrate
 - 4–7 µm electroplated Sn
 - Sn surface grains 2–5 µm in diameter



Experiment 2: Results

Exposure	Mean Density \pm STD (whisker/mm ²)	Mean Length \pm STD (μ m)	Maximum Observed Length (μ m)	# whiskers measured
TC	13 \pm 8	10 \pm 4	33	53
ETH	13 \pm 12	11 \pm 4	34	74
TC – ETH	24 \pm 24	11 \pm 4	30	97
ETH – TC	12 \pm 12	12 \pm 4	24	81
TC – ETH – TC	5 \pm 13	17 \pm 12	39	23
ETH – TC – ETH	11 \pm 12	11 \pm 4	21	62
Ambient 180 days	42 \pm 18	18 \pm 10	61	240

Little distinguishable difference between any of the exposures and ambient

Experiment 2: Correlation Between Sequential Tests

- ANOVA analysis of whisker density and length between different sequential tests

First number: density. Second number: length

1 = identical. 0= different

	TC	TH	TC-TH	TH-TC	TC-TH-TC	TH-TC-TH
TC– end of test		11	11	11	00	11
TH– end of test			01	11	00	11
TC-TH– end of test				01	00	01
TH-TC– end of test					00	11
TC-TH-TC– end of test						10
TH-TC-TH– end of test						
Ambient 44 days	00					
Ambient 149 days		00				
Ambient 168 days			00			
Ambient 180 days				00	01	00

Whisker Thickness and Correlation to Length

Questions:

- What are the variations in whisker thicknesses?
- If a correlation exists between whisker thickness and whisker length, can the diameters of whiskers be measured at some point before they reach their growth saturation, and estimate the maximum length it would grow to?
- Is it possible that the amount of Sn supplied to each whisker growing on a single surface is somewhat the same?

Whisker Thicknesses

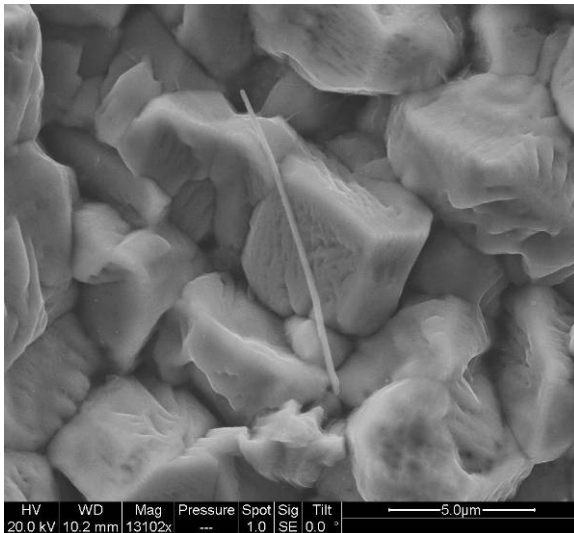
- Whisker thicknesses historically cited as sub-micron to about 10 μm
- Thickness is an important factor in

- Fusing current
$$I_{\text{melt,vacuum}} = \left(\frac{2\sqrt{Lz}T_0}{R_0} \right) \cos^{-1} \left(\frac{T_{\text{amd}}}{T_{\text{melt}}} \right) = \left(\frac{2\sqrt{Lz}T_0}{4\rho L / \pi d^2} \right) \cos^{-1} \left(\frac{T_{\text{amb}}}{T_{\text{melt}}} \right)$$

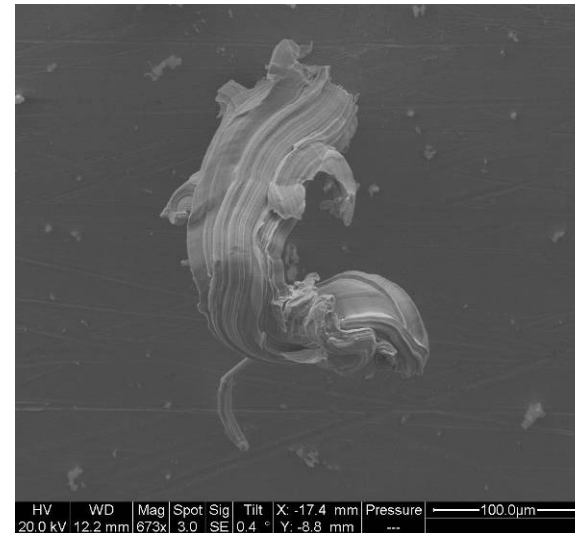
- Conformal coating penetration – calculating buckling force

$$F_B = \frac{\pi^2 EI}{(KL)^2} \approx \left(\frac{\pi^3 E}{32} \right) \left(\frac{d^4}{L^2} \right)$$

200nm thick



70 μm thick



Description of Specimens Used for Thickness and Length Data Gathering

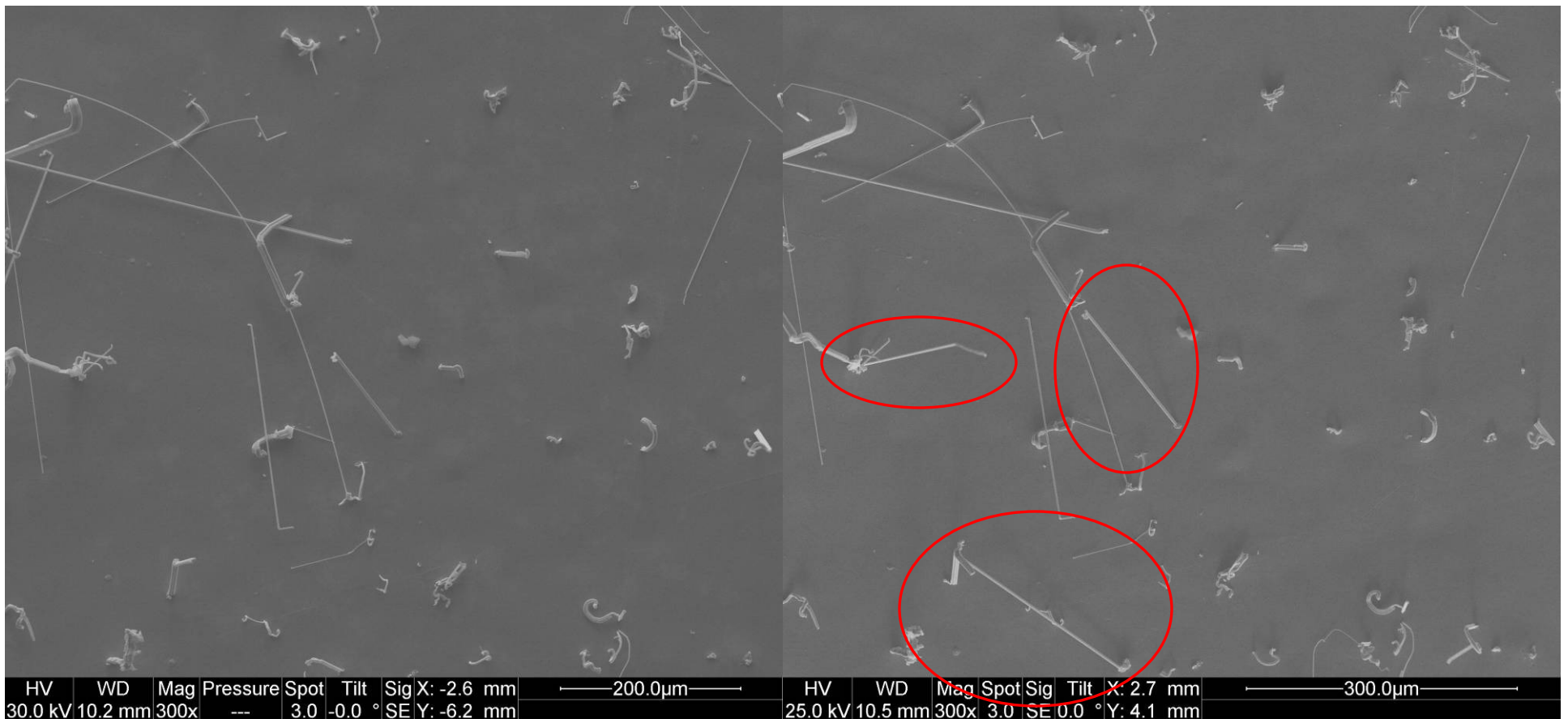
- Set 1
 - Gathered from specimens used in Experiment 1 (2.5yrs in ambient + 1000TC + 2 months ETH)
 - Whisker growth in this case considered as ‘induced’ by environmental exposure
- Set 2
 - Gathered from Sn-plated Brass specimen stored in ambient environment for ~11 years
 - Whisker growth has not completely saturated yet

Set 2 for Whisker thicknesses: Growth Not Saturated

Comparing tin whisker growth on specimen at 9.5yrs and 11 yrs of ambient exposure

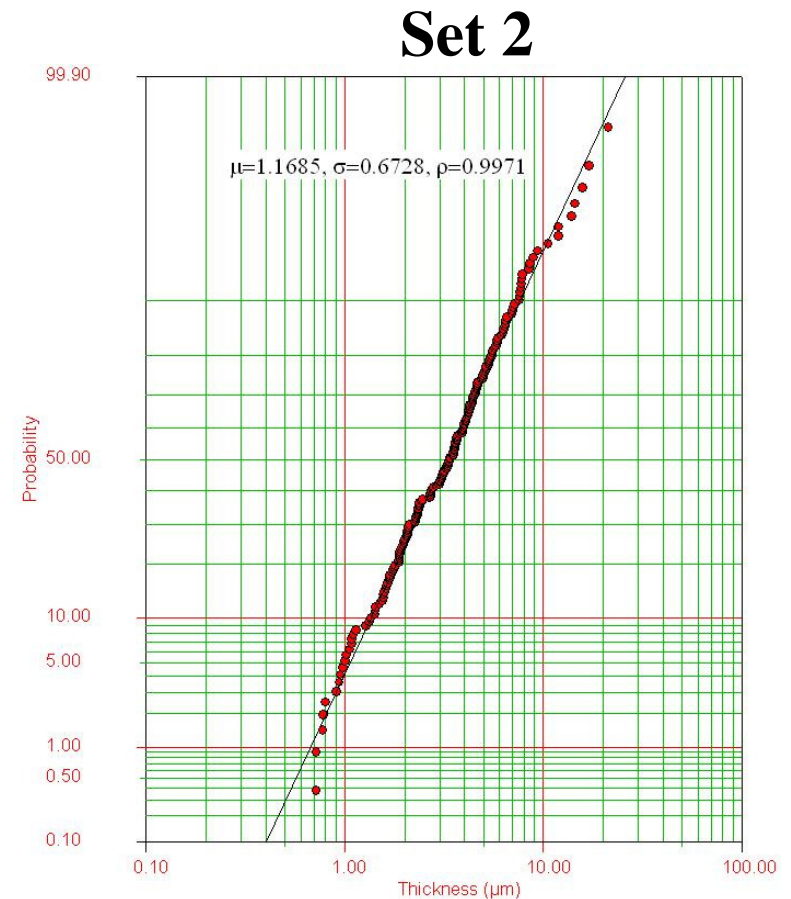
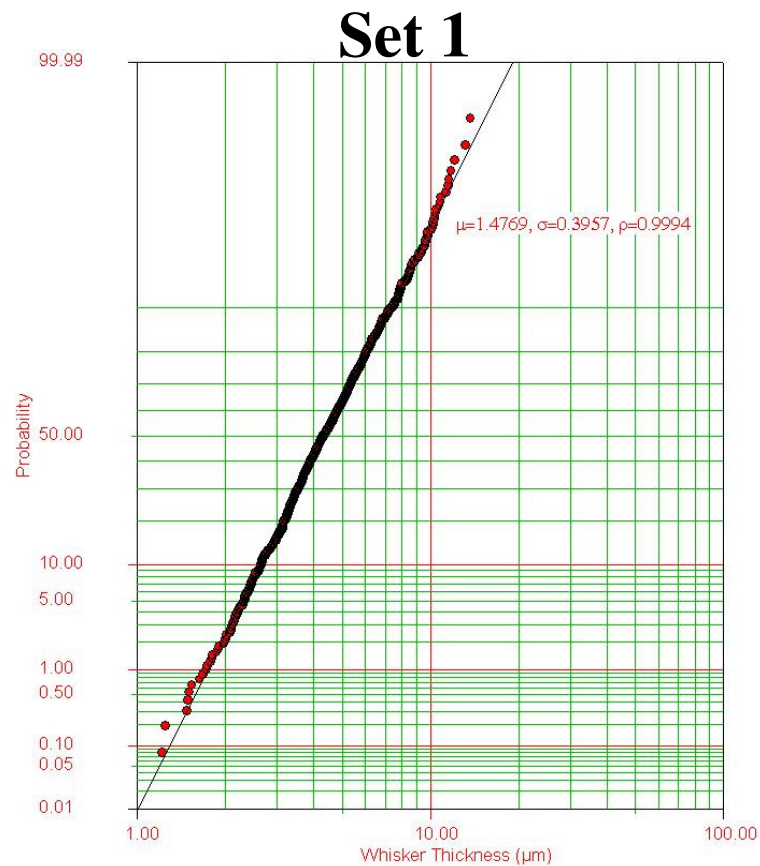
9.5yrs

11yrs



Whisker Thicknesses Distributions

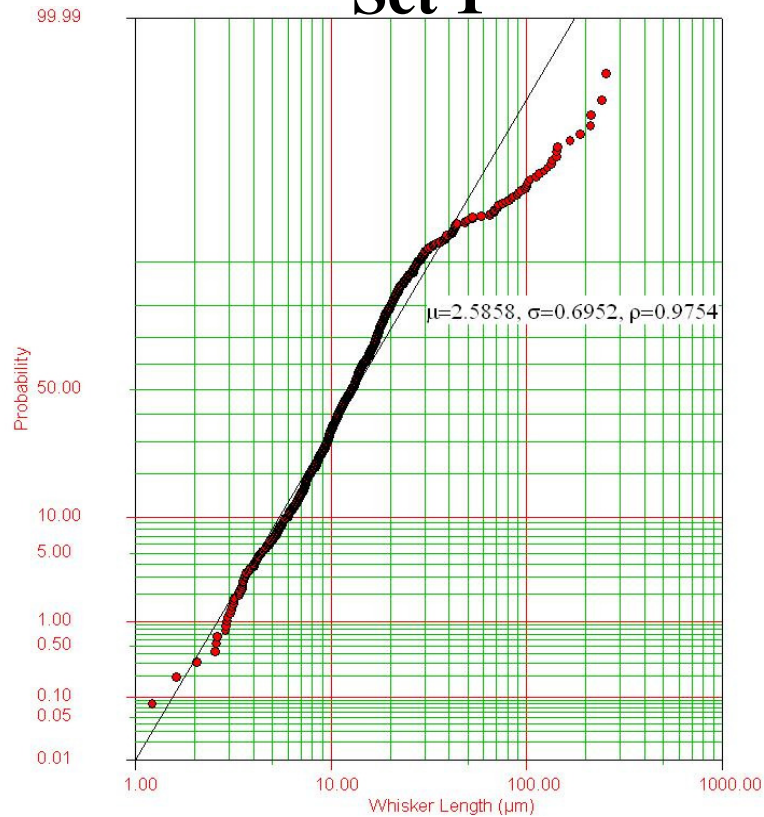
Whisker thicknesses closely followed lognormal distributions for both sets



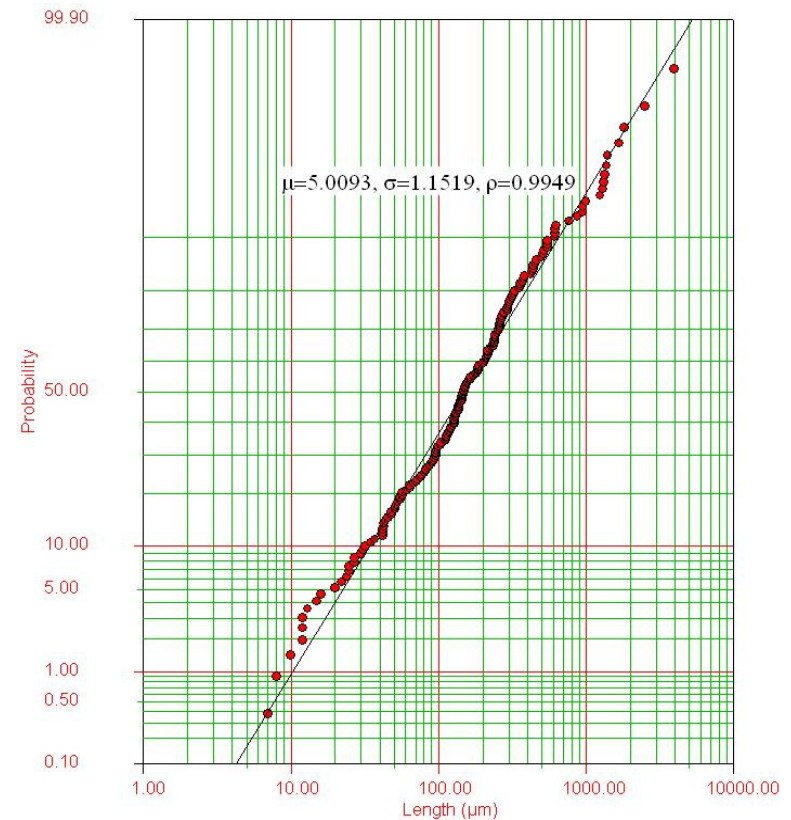
Whisker Lengths Distributions

Whisker lengths closely followed lognormal distributions for both sets

Set 1

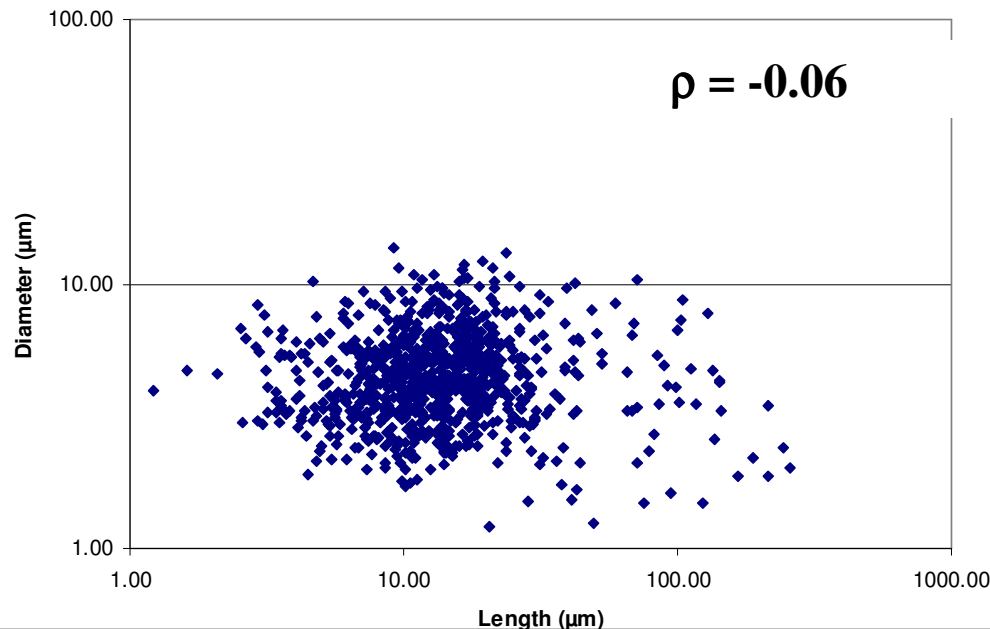


Set 2



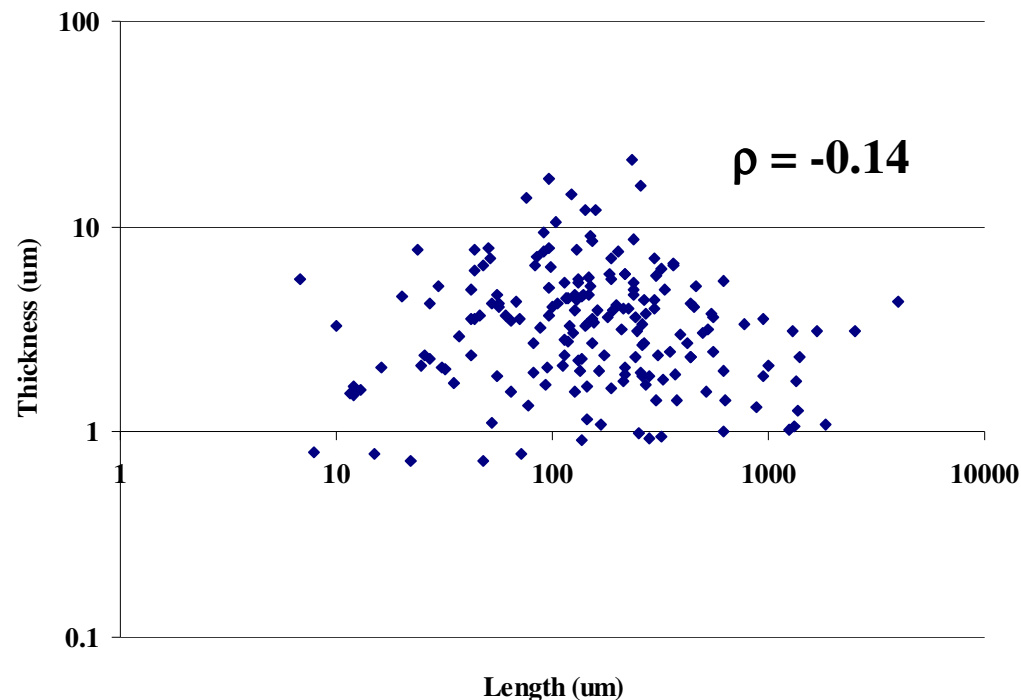
Growth Correlation: Set 1

- NO CORRELATION found (correlation coefficient -0.06) between whisker length and thickness, from data collected at the end of the test
- Attempts made to see if correlation would exist, if data is separated into subgroups, NO CORRELATION found in any of the cases
 - Underlayer separation (presence of Ni or none)
 - Separating by finish thickness
 - Correlation within individual specimens
 - Correlation for whisker with length:thickness ratios of 4:1 or greater



Growth Correlation: Set 2

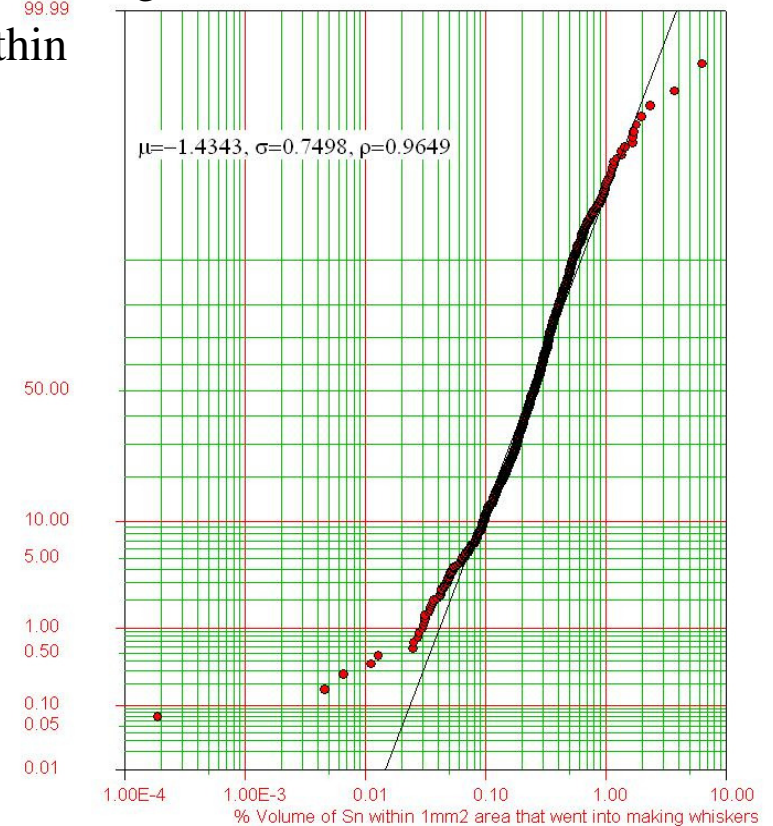
- No correlation found between whisker length and thickness after 11 years of ambient storage
- This indicates that
 - it is not possible to predict ultimate whisker length based on whisker thickness
 - the amount of Sn incoming to each whisker is not same



Volume of Sn in Whiskers

- Although material has went into Sn whiskers, no depletion of Sn is observed on the surface
- To evaluate how much Sn has been used up in making of whiskers in comparison to total volume of Sn within a given area, 1000 areas of 1mm² each were simulated with whiskers modeled through parameters described below (gathered from Set 2 data)
- Results indicate that median % of Sn available within 1mm² area used up in whiskers is 0.24%
- This agrees with lack of visual depletion of Sn

	mean	STD
Density (Normal): whiskers/mm ²	35	12
Length (Lognormal): μm	286	446
Thickness (lognormal): μm	4	3.04

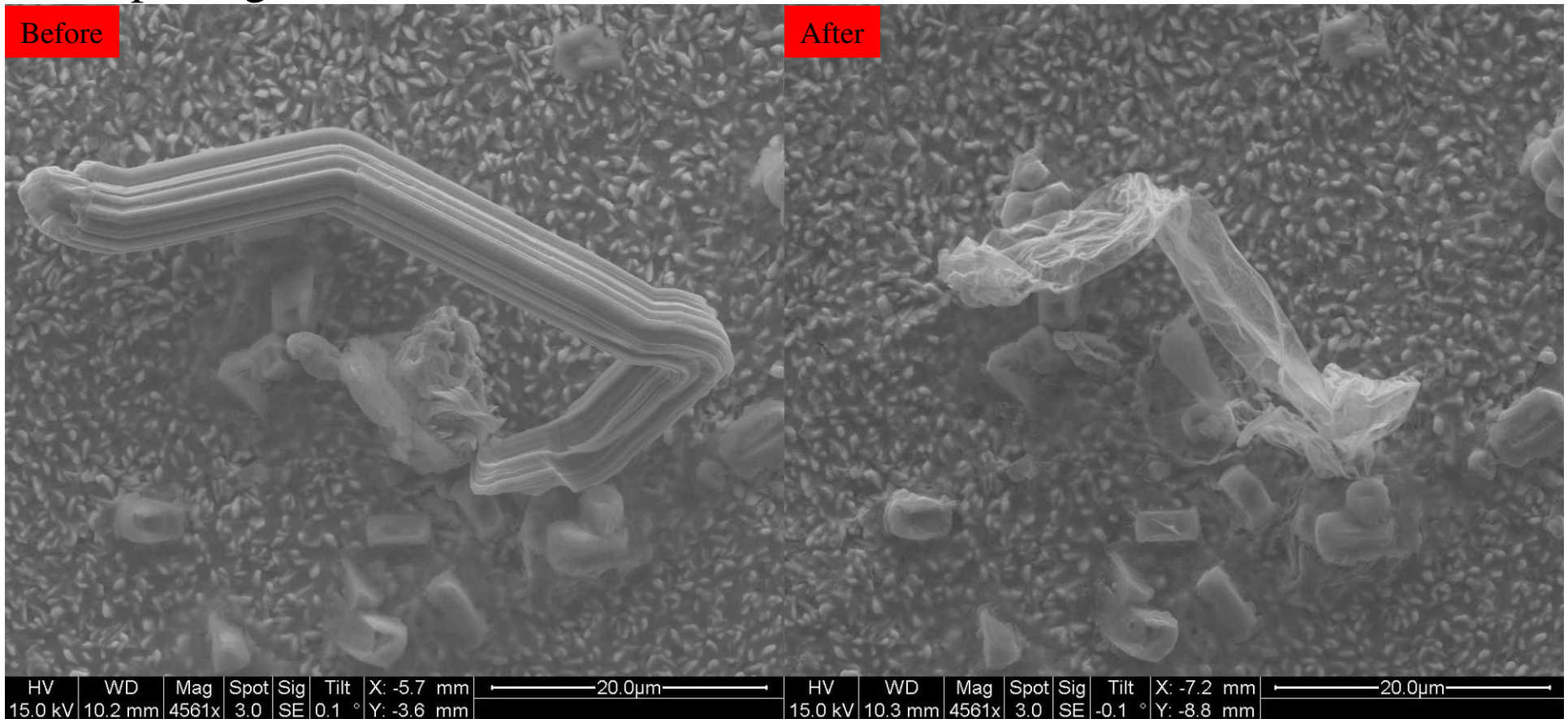


Tin Oxide Shells

Taking a look at possible useful application of tin whisker oxides as gas sensors

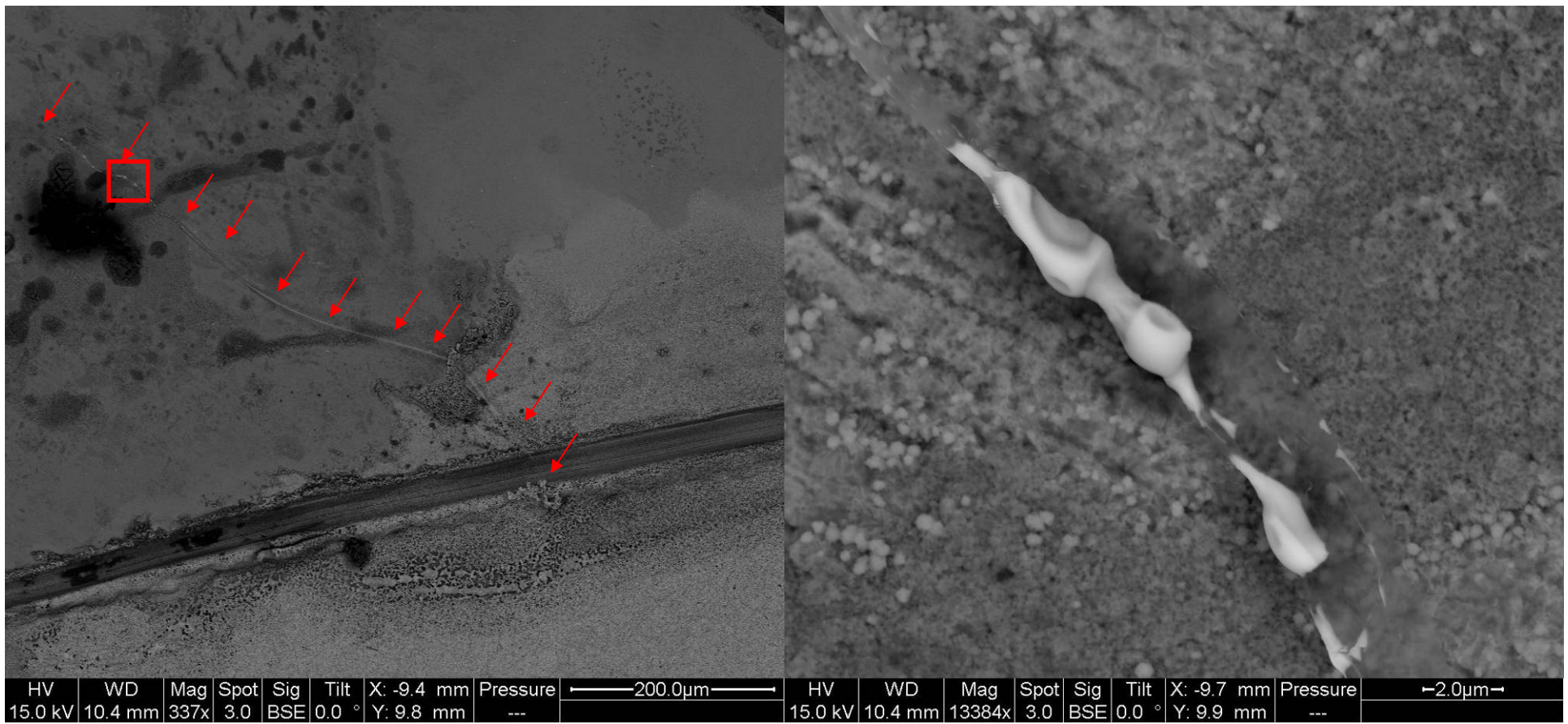
Whisker Melting

- Sn-plated Brass heated to 280°C (kept for 20min)
- Whiskers melted leaving behind “shells”
- No change in surrounding surface, suggesting that Sn expunged into plating



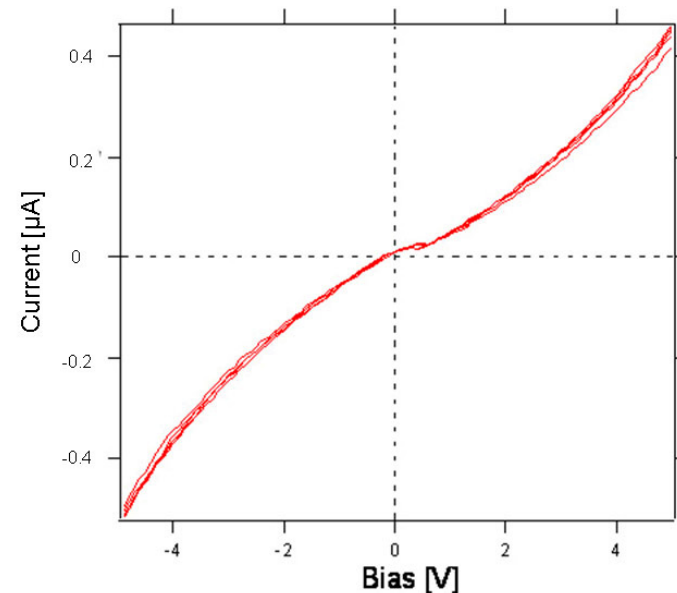
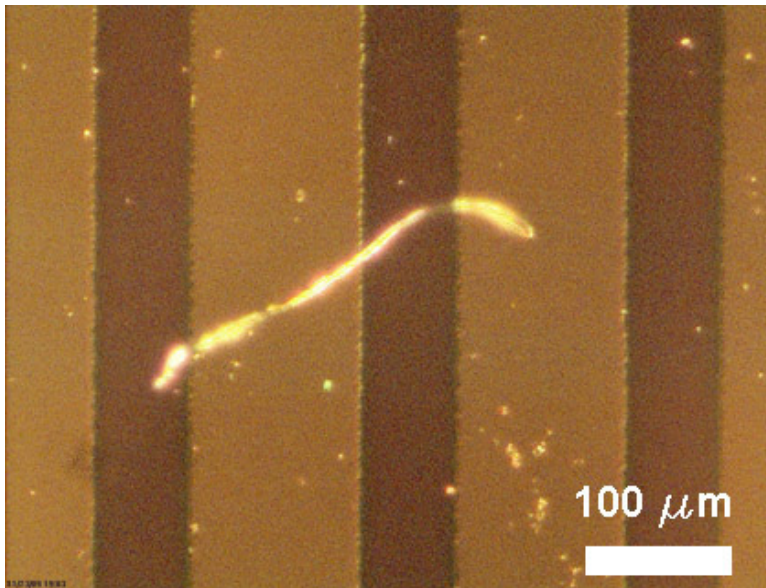
Melting Whiskers: Sn Beading

- Sn-plated Brass heated to 260°C for 15min. Whisker (~500μm) drained of Sn
- Some beaded-up Sn in the “shell”, while rest of the “shell” is empty
- This confirms Sn draining down the length of whisker to the plated surface



Potential Benefit

- Metal oxides quasi-one-dimensional structures are of interest for gas sensing technology
- Upon surface reaction with gas, a semiconductor metal oxide is transforming to create a conductive channel with significantly smaller resistance as compared to semiconductor
- Current-Voltage characteristic measured for a whisker that has been expunged of all metal – results are similar to what has been shown for tin-oxide nanowires [1]



[1] A. Kolmakov, Y. Zhang, G. Cheng, M. Moskovits, "Detection of CO and O₂ Using Tin Oxide Nanowire Sensors", *Advanced materials*, Vol. 15, No. 12, June 2003

Conclusions

Sometimes

adverb

Tin

noun

Whiskers

verb

Conclusions

- As currently implemented, environmental exposure whisker tests may under-predict, over-predict, or have little distinction from whisker growth in ambient
 - In addition, Ni underlayer does not act as a whisker mitigator
 - Use of sequential environmental exposure does not aid in whisker growth acceleration
- Whisker thicknesses follow lognormal distribution
 - No correlation exists between whisker length and thicknesses
 - Volume of tin used up in whiskers is only a fraction of available tin in the area, where long-range diffusion may act
- Preliminary results on oxide shells left behind by whisker melting showed similar characteristics to tin-oxide nanowires
 - They have a potential for becoming a new way of making carbon monoxide gas sensors

Contributions and Future Work

- Presented a reliable method for whisker length measurement
- First to document whisker thickness (diameter) distribution
- Contributed whisker parameter data
 - Whisker length, density, thickness, and growth angle distributions
- Demonstrated that existing environmental standards are not reliable in predicting long-term whisker growth
 - Sequential environmental tests can not be used consistently to increase whisker growth
 - Work in identifying what parameters contribute more to whisker growth while going through environmental exposures is required
- Identified lack of relation between whisker lengths and thicknesses
 - Whisker thicknesses demonstrated to follow lognormal distribution
- Tin-oxide shells left behind by melting tin out of the whisker, have the potential to serve as gas sensors
 - Further research needed to verify their gas sensitivity
 - Need of practical implementation in manufacturing such gas sensors

Acknowledgements

- Dr. Michael Osterman, Jay Brusse, and Dr. Henning Leidecker – for providing guidance and support throughout this work
- Lei Nie and Vidyu Challa – for the positive spirits and putting up with me
- Visiting scholars: Axel Theis, David Levi, Alexander Heronime, Choy Wai Man (Yonniss), and Dr. Tadahiro Shibutani - for help along the way
- Dr. Andrey Kolmakov and his group at South Illinois University, for their enthusiasm in tin-oxide research, and for timely providing me with preliminary experimental measurements
- And most of all, I thank my parents and family for their endless love, understanding, and support