

A Metal Finisher's Evaluation of Commercially Available Lead-Free Solder Coatings

*Michael J. Brouillard, HHEM, HH Sumco, Indianapolis, IN
Raymond O. England Ph.D., HHEM, Handy and Harman Electronic Materials Corp.,
East Providence, RI*

Environmental and toxicity concerns regarding lead-bearing solder finishes have lead the electronic industry to consider alternate solderable coatings. When the lead-free debate has been finalized a solderable, whisker-free, lead-free substitute will have to be chosen. This paper will characterize several of the commercially available lead-free tin finishes while utilizing current tin/lead systems as reference standards. Evaluation of the coatings will include thermal aging at 150°C while monitoring the effects of thermal/mechanical stresses as they relate to solderability, contact resistance and whisker formation. Scanning electron microscopy and energy dispersive spectroscopy are used to characterize the microstructures of the various systems.

For more information, contact:

Michael J. Brouillard, CEF-SE
Handy and Harman Electronic Materials
HH Sumco Inc.
1351 South Girls School Rd.
Indianapolis, IN 46231

Phone: (317) 241-7600
FAX: (317) 248-2352

Raymond O. England, Ph.D., CEF
Handy and Harman Electronic Materials
HH Electronic Materials Group
231 Ferris Ave.
East Providence, RI

Phone: (401) 434-6543
FAX: (401) 438-6417

Introduction

Because of its unique combination of properties, solderable tin/lead surface coatings have been standard in the electronics industry for many years¹. Today, tin/lead solderable coatings are widely used for electronic components ranging from heavy load automotive connectors, to high technology integrated circuits. In the near future, government legislation will mandate that solderable surfaces be free from lead. The European Community (EC) has already set the date of 01/01/2004² for compliance with a lead free directive. At the suggestion of local governments, Japan, and the Pacific Rim countries have already begun voluntary reductions in the use of leaded products. It is expected that by 2001, lead free policies will be adopted here as well.

The pending elimination of such a prominent product has led to much research in the area of lead free solderable coatings. Many alternative tin and precious metal systems such as SnCu, SnAg, SnZn, SnBi, SnSb, SnIn, SnAgCu, SnAgCuSb, SnBiSbAg, PdNi, and PdNiAu have been proposed. These coating systems are viable replacements for tin-lead coatings, but at a cost premium. This paper evaluates four commercially available pure tin coatings. These coatings are listed in Table 1. They are fairly well known and in most cases less expensive than the systems mentioned earlier.

Sample #	Solderable Coating
1	90/10 Matte Tin-Lead
2	Matte Tin A – Typical
3	Bright Electroplated Tin
4	Matte Tin B – Proprietary
5	Reflowed Electroplated Tin

Table 1

In this paper, standard connector and integrated circuit component performance criteria are used to evaluate the five candidates.

The ability to efficiently carry a load as a function of time and duty conditions is critical for most electronic components. For instance, in many critical applications the automotive industry is reluctant to specify coatings that exhibit contact resistance values in excess of one milliohm over the expected service life [300g normal force]. Based on comments from industry sources; values of around five milliohms cause

great concern, and ten milliohms is unacceptable. An accelerated test to evaluate change in contact resistance as a function of time at 150°C (302°F) was completed in accordance with ASTM standards 667 and 539. Growth of both Cu₃Sn and Cu₆Sn₅ intermetallics occurs via solid state diffusion of copper from the base alloy into the tin coating^{3,4}. The conversion of the tin coating from the interface of the base copper toward the surface of the coating proceeds at some rate, which is a function of time, temperature, and coating chemistry⁵. The free tin is consumed as the intermetallic reaction proceeds with time and as such is a decreasing function. The point at which total conversion of the free tin to intermetallic is achieved results in drastic increase in the surface contact resistance. X-ray Fluorescence (XRF) techniques are used to determine free Sn consumption as a function of aging time at 150°C.

Most integrated circuit components must be solderable (and wire bondable) at the assembly point. This seemingly mundane requirement takes on critical significance due to the component value added at the point when soldering is required. The complex geometry of the leadframe has been stamped from strip material using large high-speed hydraulic presses and part specific tooling. Additionally, multiple electroplated metals such as nickel and/or tin/lead have been applied to various locations, and spot coatings of precious metals such as gold and palladium requiring selective plating tooling have been imparted to critical areas. Additionally, it is well known that shelf life is an issue with many solderable tin/lead coatings⁶. Solderability is evaluated in an accelerated test as a function of aging time at 150°C using METHOD 2003 of MIL STD 883.

In this study, tribological performance is evaluated in a self-mated, cyclic sliding wear experiment. For some solderable surfaces, particularly in automotive and digital signal processing applications, high density pin connectors are beginning to require low insertion forces. As the pin density continues to increase, this will be of increasing concern as the frictional force required to properly insert the pins into the receptacles will become so high as to require specialized tooling⁷.

Pure tin coatings are known to "grow" whiskers as a function of many component variables such as brightener additions, substrate metallurgy, compressive stress level, and thickness⁸. Whiskers are undesirable in high device density IC applications as they can

achieve sufficient length to bridge across individual components causing a short. The candidate materials were subjected to various levels of compressive stress in bending at room temperature to assess the propensity for whisker development.

Sample Matrix

The base metal utilized for this study was an alloy UNS C42500 having a size of 0.025cm x 3.60cm (0.010" x 1.417"). Alloy 425 was chosen for this study due to its popularity in the connector industry. All the samples were plated on a production strip plating machine to replicate typical production conditions. An underplate of copper was utilized on each sample, the thickness of the copper underplate was 1.25 micro-meters (50 micro-inches) nominal. The addition of the copper underplate was incorporated in an effort to reduce/eliminate the effects of base metal diffusion which may effect the properties of the solderable coatings over time. The solderable coatings tested in this study can be seen in table #1.

The coatings found in table #1 are not intended to represent all of the lead-free solder coatings available to the typical metal finisher, but does include many of the systems currently in production. At the time of this paper several interesting alloys were being developed but were not ready for testing prior to the papers deadline. Some of the more interesting alloys include copper-tin and copper-tin-silver alloys.

The reasoning behind the coating selection found in Table #1 are as follows: 90/10 tin/lead was chosen as the reference coating. The alloy composition was chosen for it's low lead concentration. Lead is used in solder coatings for several reasons. One of which is to lower the temperature at which tin-lead melts. Secondly lead is used as an additive to inhibit whisker growth. When low solder temperatures are not of primary importance, small amounts of lead can be applied to the tin-lead alloy to ensure whisker-free deposits. This study will focus on the lower lead containing tin-lead coatings, where the relatively higher melting point is not paramount to over-all functionality.

The bright-electroplated tin deposit (sample #3) was included in the matrix as a standard of sorts. Bright tin has long been known for it's tendency to grow whiskers on plated/stamped product. The term "whisker" denotes a very fine, hair-like single crystal that forms from certain materials ... tin being one of

them. Whiskers can form spontaneously under certain conditions. Tin whiskers are typically around 1 micrometer in diameter and can grow to several millimeters in length. In the case of tin-plated electronic components, whisker formation can, by bridging between connectors, cause short-circuits and thus total failure of an electronic assembly. Whisker growth is largely due to mechanical stresses, in tin coatings. Stresses can arise by codeposition of organic brighteners or by their breakdown or reaction products. The organic additives used in bright tin plating can favor co-deposition of hydrogen. They tend themselves to be codeposited and thus increase deposit stress.⁹ Whiskers have also been noticed on matte tin deposits where organic levels were low, but the propensity for whisker growth is far greater on bright-electroplated tin coatings.

Reflowed electroplated tin (sample #5) was included in the study from a standpoint of attaining a bright finish similar to bright-electroplated tin. Reflowed tin, unlike the bright-electroplated tin, does not have any of the inherent stresses found in organically brightened coatings. It is not unusual for bright tin electroplating solutions to contain 10 times more organics than matte tin plating baths.¹⁰ The lower organic level combined with the stress relieving effects of the melt/quench dynamics allow the deposit to have a bright appearance without the inherent stress and brittleness associated with bright tin plating.

Two types of matte electroplated finishes were also included in the matrix. The first deposit is a "standard" methane sulfonic acid (MSA) based plating system (sample #2). This sample was included to test the theory that the simple fact of lowering the organic concentration would lower internal stresses enough to eliminate whiskering. Sample #4 is a proprietary MSA matte tin plating system formulated and marketed as a lead-free, whisker-free deposit.

Experimental Procedures

Sample Geometry

Sample geometry for the contact resistance, solderability, and free tin experiments are nominally 20.32 x 0.025 x 3.60cm (8.0 x 0.010 x 1.417 inches). Each sample is punched with a 0.317cm (0.125 inch) diameter hole to aid in furnace aging.

Contact Resistance

Contact resistance is evaluated using the practices, outlined in ASTM specifications 667 and 539^{11,12}. Method C, Dry Circuit Testing, is used in this study. A power supply which delivers an open circuit voltage of 20 millivolts maximum, with a short circuit current of 100 milliamps is used. The test apparatus is shown in Figure 1. It consists of: 1) A programmable servo motor controlled gold probe (for Z axis stroke control), having a radius of 3.2 mm, 2) A sample stage with manual X, Y, Z position control, 3) A Keithley 580 micro-ohm meter which supplies the small open circuit voltage and measures the sample contact resistance, 3) An in-line Omega LCF series tension/compression load cell, and 4) An Omega type 41 controller. Prior to and after each measurement, the contact resistance of a pure gold standard coupon is checked to assure it is within the range 0.75 to 1.5 milliohms at 25 g load. Three measurements of the gold standard are made before and after test, all of which must be within the specified range for the test data to be valid.

The contact resistance for the candidate alloys is measured as a function of aging time at 150°C. Exposure times are 0, 240, 480, 720, and 1000h. Contact resistance data values at each exposure condition at 300g normal load are the reported median of ten random measurements taken along the approximate specimen centerline. The probe is brought into close proximity with the specimen surface, and then as contact is made, the load is increased to the test value of 300g. There is a slight relaxation of applied load over the first twenty seconds as the system stabilizes. After stabilization, the test load is slowly increased back to 300g, at which point the resistance value is recorded.

Solderability

Solderability is evaluated in accordance with MIL-STD 883, Method 2003. The test apparatus is a Kester 1000 automated solderability tester and is shown in Figure 2. The solder cycle is as follows: 1) Dip in flux, and dwell for 5 seconds, 2) Move out of solder to position above the solder pot and dwell for 10 seconds, (Note: as the specimen moves to position above the solder pot, a mechanical wipe of the solder surface is made thus assuring a slag-free dip), 3) Dip in Solder and dwell for 5

seconds, and 4) Retract specimen and return to home position. The solder flux is non-activated N-100 (rosin/isopropyl alcohol). The solder used is a 63Sn/37Pb eutectic alloy. Test specimens are placed vertically in the holder, and retained using a face plate and screw. The flux is maintained at room temperature, and the solder pot is held at 245°C.

Solderability is characterized as a function of aging time at 150°C (302°F). Aging times are 0, 8, 24, 48, 72, 96, 120, 144, and 168h. The failure criteria for solderability is here defined as coverage of less than 100%.

Free Tin Analysis

Free Sn is determined as a function of aging at 150°C. A Veeco Instruments X-ray Fluorescence (XRF) unit. The sample conditions are 10-second excitation at 50KeV accelerating voltage, with a 0.152mm (0.006 inch) diameter beam. Three random readings are made of the specimen surface along the approximate centerline. The mean of these readings is reported. The specimen is then placed in an acid tin-stripping bath to remove the surface tin which has not been converted to the copper/tin intermetallic phases Cu_3Sn and Cu_6Sn_5 . Again, three XRF readings are taken. The difference in these two readings corresponds to the thickness of unconverted tin. Aging times at 150°C are 0, 8, 24, 48, 72, 96, 120, 144, 168 240, 480, 720, and 1000h.

Friction and Wear

Friction and wear performance is evaluated using a self-mated 10 cycle reciprocating sliding wear experiment. The schematic representation of the friction and wear tester and a photograph is shown schematically in Figure 3. Two specimen configurations are needed for the test; a slider strip, and a profiled strip. The slider strip is nominally 20.32 x 0.025 x 3.60cm (8.0 x 0.010 x 1.417 inches). The profiled strip has a die formed 3.2 mm diameter hemisphere in its geometric center. Prior to forming which imparts the hemisphere and a u-shaped geometry, the profiled strip has the dimensions 0.825 x 0.635 x 0.025 cm (0.325 x 0.25 x 0.010 inches). The long axis of the specimen is in the transverse orientation relative to the original plated strip. Thus the direction of sliding for the profiled strip is longitudinal relative to the original strip. The direction of

sliding of the profiled specimen on the slider plate is transverse relative to the original strip. A normal load of 67.3g is applied to the profiled strip. Each of the ten test cycles consists of a forward and backward relative movement of the slider plate and profiled specimen. The travel distance for each half cycle is 1.9cm (0.750 inches) at a travel speed of 1mm/s¹. Data is collected via a link between the Omega controller and the R/S 232 port of a Personal Computer. Data is acquired at a rate of 10 Hz. All friction and wear tests are conducted at ambient conditions.

Whisker Growth

Propensity to grow Sn whiskers was evaluated at both room temperature and 50°C. For the room temperature experiments, specimens were subjected to two levels of compressive stress in bending. The expression for bending stress as a function of radius of curvature, elastic modulus, beam thickness, and yield stress is used¹³. This expression is

$$\rho = \left(E \left(\frac{h}{2} \right) \right) \sigma^{-1}$$

where:

ρ = Bending Radius (in.)

σ = Yield Stress (psi)

h = Beam Thickness (in)

E = Elastic Modulus (psi)

The reported range for the yield stress of tin deposits was between 4 and 20 ksi. The elastic modulus is approximately 6×10^6 . 10 and 20 ksi applied stress levels were selected as stress levels for testing. Using these parameters, and the thickness of the coated specimen, values for radius of curvature were calculated to be 5.72cm and 11.43cm (2.25 and 4.5 inches, respectively). These values were used to determine the appropriate diameter for two test "circles" for each material. Appropriate lengths of strip were cut from each coil, and projection spot welds were used to splice the ends together. A slight overlap was used to assure adequate weld strength. The concave surface of each specimen is of course in compression, and is where whisker growth is expected to occur. Samples of the bright-electroplated tin were also baked at 50°C to test the theory that "whiskering occurs fastest around 50°C, the recrystallization temperature of tin."¹⁴

Results

Contact Resistance

Figure 4 shows the change in contact resistance for each alloy as a function of aging time at 150°C. As expected, contact resistance increases for all alloys as a function of time at temperature. However, only the 90/10 tin/lead and the matte tin "B" materials stayed at or below the critical value of 1 milliohm through 420 hours. Overall, the 90/10 tin/lead demonstrated the most resistance to thermal aging followed closely by matte tin "B". The bright sulfate tin and matte tin "A" behaved essentially the same both reaching values of approximately ten milliohms at 420 hours. After 420 hours aging time the reflow tin increased to a contact resistance value of around 1 ohm.

Solderability

Figure 5 graphically represents the results of solderability test. The matte tin "B" sample retained 100% coverage for 96 hours. The reflowed tin was the next most resistant to solderable surface deterioration at 72 hours, followed by both the matte tin "A" and 90/10 tin/lead at 48 hours, and finally, the bright electroplated tin at 24 hours. Figure 6 shows representative photomicrographs of the solder surface as plated (zero hour exposure), and at the failure condition for each material.

Free Tin

Figure 7 shows the trend in consumption of free tin as a function of exposure time. As expected, the rate of intermetallic consumption is high for all coatings. The matte tin "B" sample showed the most resistance to intermetallic conversion retaining roughly 1.0 micro-meters (40 microinches) at the 420-hour aging condition. The reflowed tin and 90/10 tin/lead samples performed essentially the same, having values of around 0.5 micro-meters (20 microinches) after 168 hours. With continued aging the tin/lead was somewhat variable, and reached a value of just over 0.5 micro-meters (20 microinches) at 420 hours. The reflow tin was less variable, and had just below 0.5 micro-meters (20 microinches) at 420 hours. The bright and matte tin "A" samples both had free tin

values approaching zero after 96 hours. Interestingly, these materials were variable, and seemed to rebound slightly with continued aging to 420 hours.

Friction and Wear

Figure 8 shows the friction coefficient results for the candidate coatings. The positive and negative values for friction coefficient (peak and trough) represent the forward and reverse frictional force generated during each cycle. Determining relative performance using the Figure 8 data format can be subjective. To resolve this issue, a single metric format is used. This metric is the median value for the array of data points collected for each test. It represents the median force required to move the specimen over the slider block for the ten cycle test. This data is shown in Figure 9. The 90/10 tin/lead and the bright electroplated tin both showed friction coefficients of around 0.6. The matte tin “A”, matte tin “B”, and reflow tin had somewhat higher values of from 0.8 to 0.9.

Whisker Growth

The plated samples were visually inspected for evidence of tin “whiskering”. All samples were pre-stressed and allowed to age in air as noted in the previous section. At regular intervals samples were visually inspected with the aid of a low power microscope (approximately 40X). Samples exhibiting any sign of irregularities on the surface of the plated strip were analyzed further via Scanning Electron Microscope (SEM). Samples of the pre-stressed bright tin were also subjected to a hot air bake at 50°C (122°F) for approximately 450 hours. Samples of the heat-aged bright tin had a propensity for increased specular reflectivity. These samples were viewed under very high magnification and evidence of whiskers was observed. The whiskers were photo documented; a sampling of these whiskers can be seen in figure 10. Many of the whiskers found on this sample appeared to have a base resembling a tree trunk, which anchored the whisker to the plated base. Figure 11, shows a rare occasion where two whiskers have grown adjacent to each other, while figure 12 can be described as a family of whiskers, showing signs of how whiskers are initially formed. The whiskers observed in this study had an approximate diameter of 3 micro-meters (150 micro-inches) and an approximate

length of 13 micro-meters (550 micro-inches). Based on the “success” of the baking trials, samples from each of the representative coatings were baked at 50°Cⁱ. Whiskers were also evident on the pre-stressed bright tin sample without thermal aging, these whiskers were relatively smaller and the population was considerably less. All other air-aged samples showed no signs of whiskers after the first 30 days. This study is on-going, progress and findings will be documented for later review.

Discussion

Sn forms a thin, dense, tightly adherent, protective native oxide SnO₂. This oxide crystallizes in the rutile structure, which is a slightly distorted nearly closest packed anion array where the coordination number for O around each Sn atom is six^{15,16}. SnO₂ is thick and dense enough to protect the surface from oxidation, but it is not so thick as to increase the surface contact resistance, or degrade solderability. So long as this oxide remains intact, the desirable electronic surface properties of the Sn coating will be maintained. Phenomena which can compromise the protective nature of the oxide layer are the growth of a copper/tin intermetallic zone via solid state diffusion of copper, and/or rapid diffusion of species such as copper, zinc, and other contaminants to the coating/oxide interface where they can subsequently oxidize. All coatings used in this study were plated onto the same base copper alloy, alloy 425. So as to provide an equal baseline condition, all alloys were given a nominal barrier layer of 1.25 micro-meters (50 microinches) copper¹⁷. Therefore, the performance difference between the candidate materials is dependent upon the rate of intermetallic zone growth, and solid state diffusion of contaminants unique to each coating.

Differences in the change in contact resistance values as a function of aging can be understood in terms of each coating's unique characteristics.

XRF analysis at the zero hour aging condition indicates that the 90/10 tin/lead and the matte tin “B” sample have the most available free tin. It is not surprising that these materials

ⁱ Authors Note: Results of this study were not completed in time to include in the paper; findings will be presented at the SurFin 2000 presentation.

have the best retention of contact resistance over time at temperature. Compared to the 90/10 tin/lead and matte tin “B”, the matte tin “A”, the electroplated bright tin, and reflowed tin specimens have lower relative initial free tin. Although solid state mixing of the copper barrier and tin coatings and subsequent intermetallic zone growth is expected for all coatings, the reflow process imparts significant heat to the coating surface. This heat input may help to jump-start the intermetallic zone growth process relative to the other candidate materials. This intermetallic jump-start may also be the reason for the relatively high contact resistance achieved at the 420 hour exposure condition. The electroplated bright tin and matte tin “A” performed essentially the same. This is not unexpected since the initial coating thickness values are very close. It is likely that the extent of intermetallic conversion and/or the amount of contaminants at the surface at 420 hours exposure is less for these materials than that attained by the reflow material. This might explain the somewhat lower reading for contact resistance versus the reflow material.

The solderability results are interesting. The matte tin “A”, matte tin “B”, and 90/10 tin/lead are similar coatings, all being matte in appearance. Yet the matte tin “B” performs much better than either the 90/10 tin/lead or the standard matte tin “A”. Diffusion of contaminants from the base metal strongly influences the ability to retain a solderable surface. As such, it appears that contaminant diffusion through the matte tin “B” coating is inhibited to a greater degree. The reflow material demonstrated the second best performance. One possible explanation for this might be that the *in situ* formation of a relatively thick intermetallic zone due to the reflow process itself might act as an initial diffusion barrier to contaminant species. Another possible explanation is that the reflow process might “anneal” the tin deposit reducing its residual stress thus increasing the activation energy barrier for contaminant diffusion. The organic brighteners used to develop the fine grain size of the bright tin also impart significant residual stress to the coating. Since more grain boundary area is available for contaminant diffusion, and since the residual stress might be expected to reduce the activation energy barrier for contaminant diffusion. It is not surprising that this material was the first to fail solderability testing.

Results for the friction coefficient testing reflect both the chemistry and surface finish of the candidate coatings. Additions of lead to tin result in a reduced friction coefficient. This is easily seen as one compares the performance of the matte finish tin/lead coating to the pure matte tin coatings. The relatively low friction coefficient observed for the electroplated bright tin coating is attributed to its high hardness, and its low surface roughness¹⁸. The higher friction coefficient for the reflow tin, matte tin “A”, and matte tin “B” is due to the combination of low hardness and relatively higher surface roughness.

The whisker growth portion of this study should be characterized as “work in progress”. This research has uncovered studies that have been analyzing tin coatings for the effects of whiskers for over 20 years¹⁹. The methods of growing whiskers utilized in this study can be characterized as the “best available practices”, understanding that more work needs to be focused on this type of testing. An unequivocal explanation of the causes and mechanisms of whisker growth has yet to be developed. However on the basis of numerous independent studies, it can be confidently assumed that whisker growth is largely due to mechanical forces²⁰. It should also be stated that many external factors could contribute to these stresses including; incubation times, temperature, atmospheric conditions, substrate effects, and deposition conditions.

Conclusion

Characterization of commercially available lead-free solderable coatings has shown significant variation in performance criteria commonly evaluated in the electronics industry. Selection of a solderable material should be made with due consideration given to the specific application requirements.

Acknowledgement

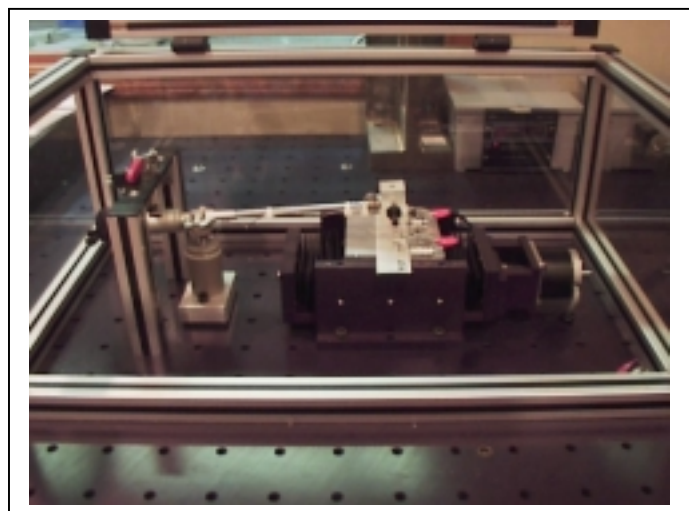
The authors gratefully acknowledge the contributions of L. Kabakian who assisted in all aspects of sample testing. Additionally, thanks to J. Fister and Dr. J. Steel of Olin Brass for many helpful technical discussions and for providing the friction and wear test data. The authors would also like to thank the American Electroplaters and Surface Finishers Society for the opportunity to publish and present this data.

**Figure 1 - Contact
Resistance Tester**



Figure 2 - Solderability Tester

Figure 3 - Friction and Wear Tester



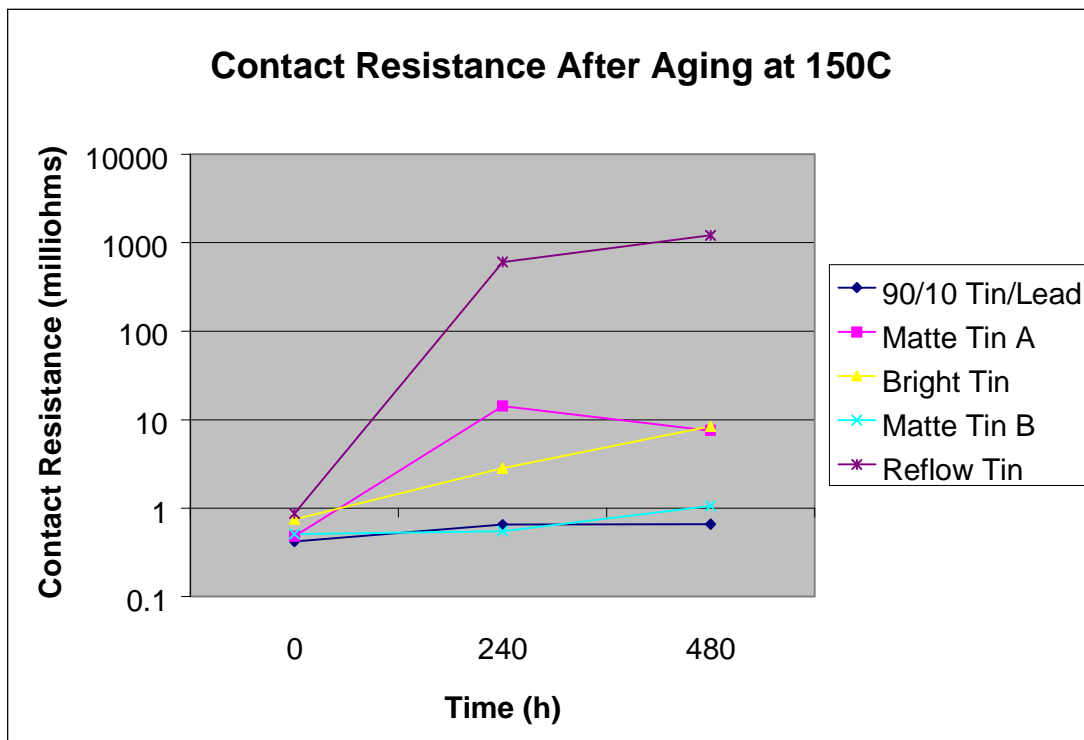


Figure 4 - Contact Resistance After Thermal Aging

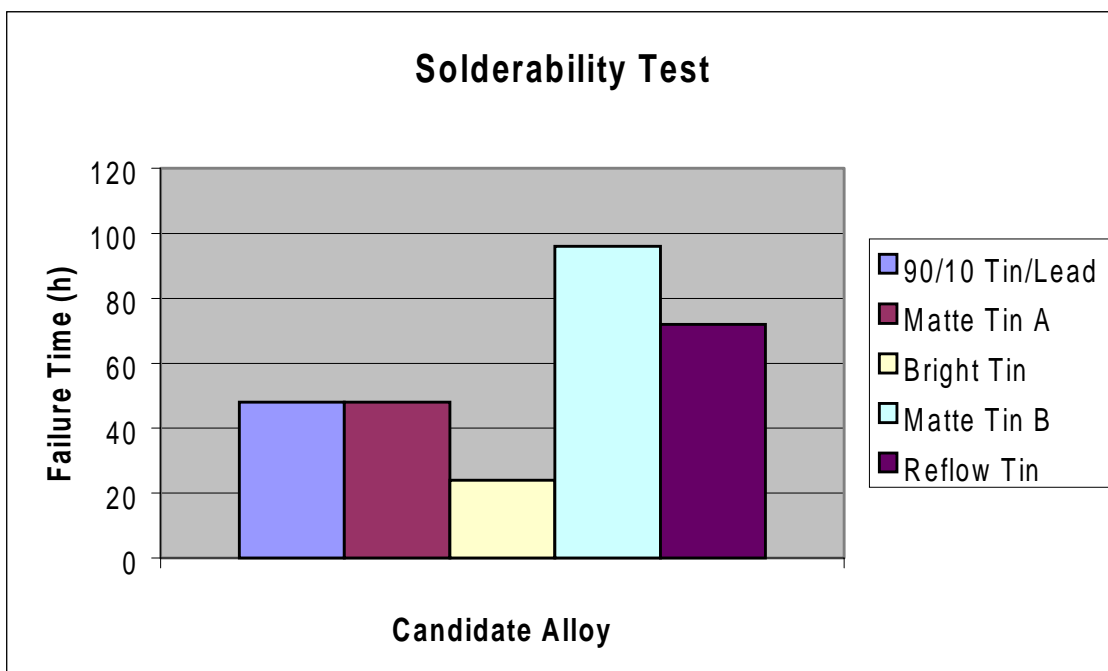


Figure 5 - Solderability Test Results







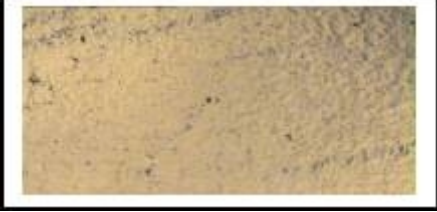



Sample	Solderability	
Identification	As-Plated	At-Failure
Sample #1 90/10 Tin/Lead		
Sample #2 Matte Tin Typical		
Sample #3 Bright Tin		
Sample #4 Matte Tin Proprietary		
Sample #5 Reflow Tin		

Figure 6 – Solderability Results

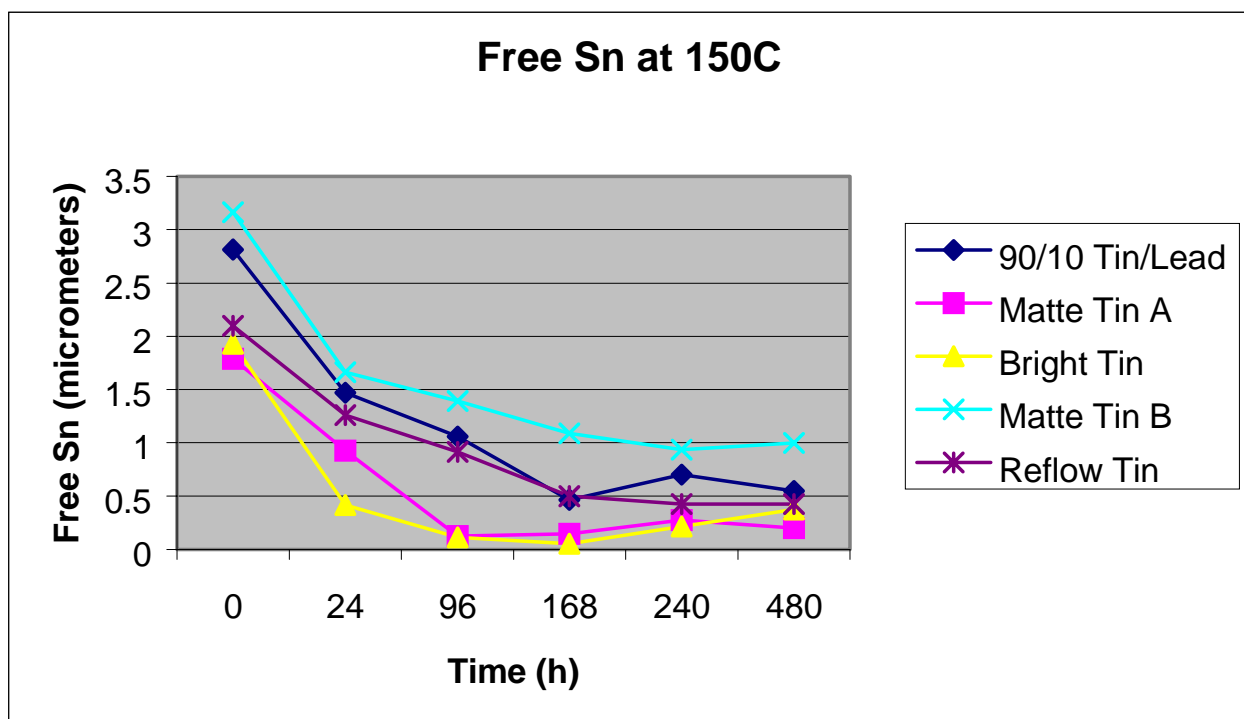


Figure 7 – Free Tin Analysis

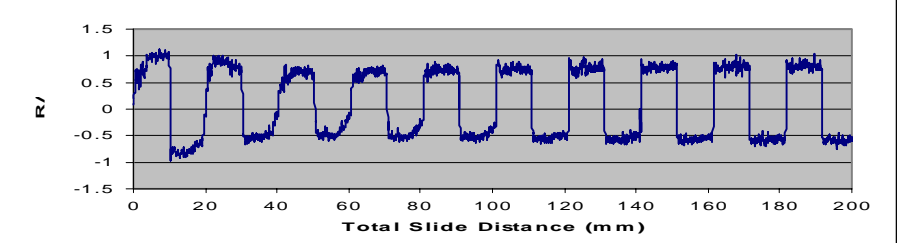
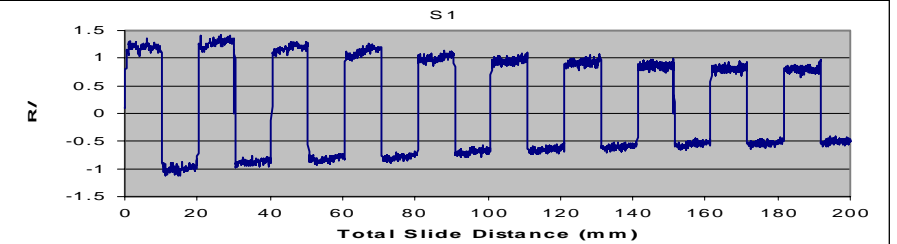
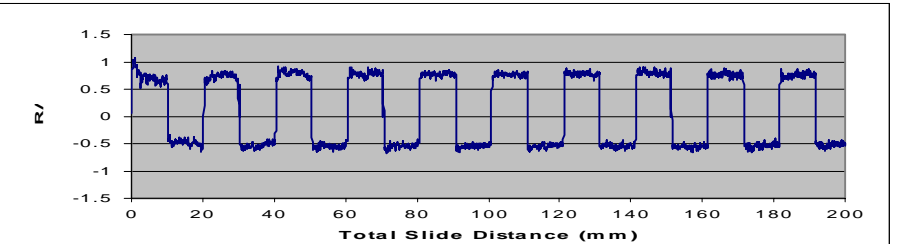
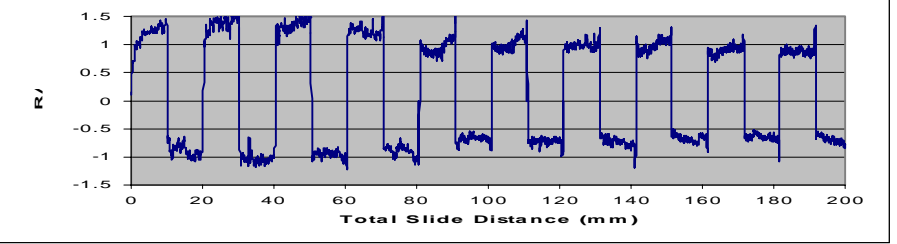
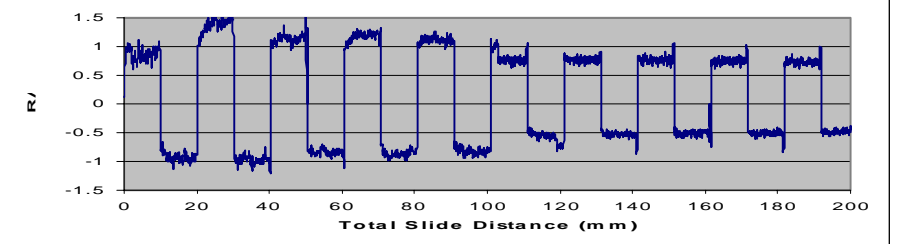
Sample	Coefficient of Friction
Sample #1 90/10 Tin/Lead	
Sample #2 Matte Tin Typical	
Sample #3 Bright Tin	
Sample #4 Matte Tin Proprietary	
Sample #5 Reflow Tin	

Figure 8 – Coefficient of Friction Results

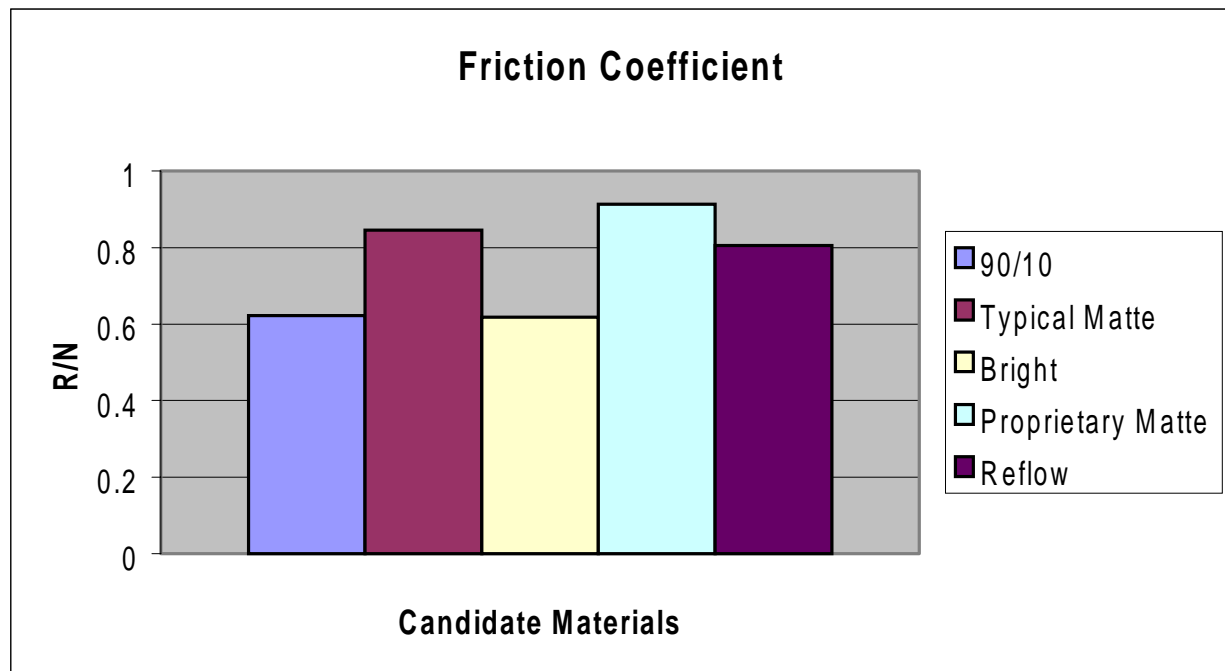
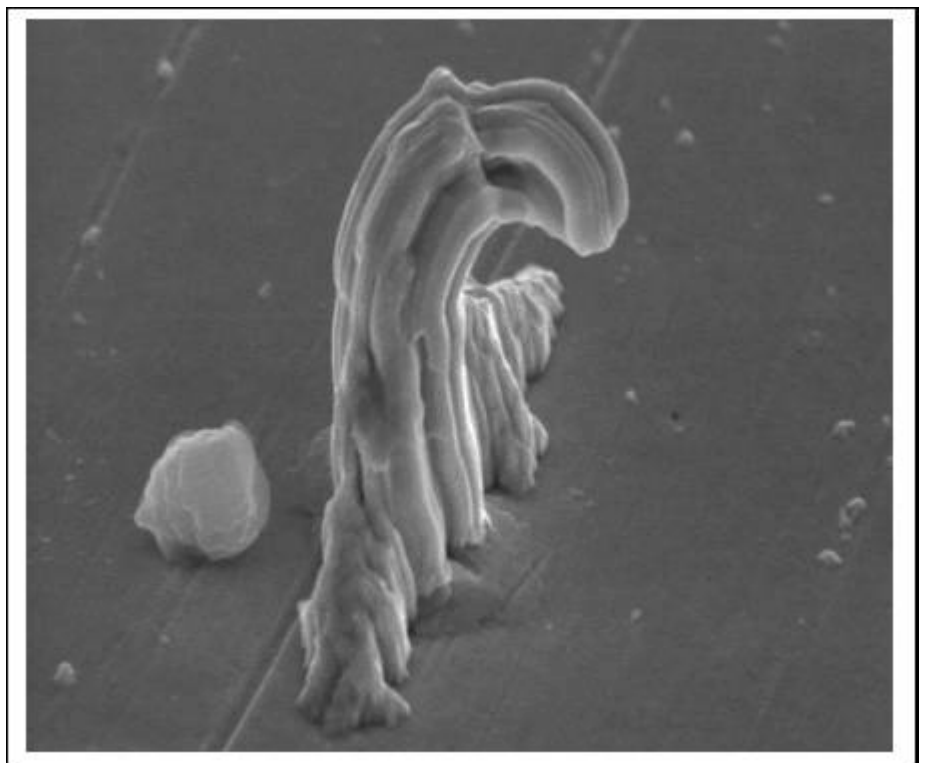


Figure 9 - Median Coefficient of Friction Values

Figure 10

Magnification = 5350X



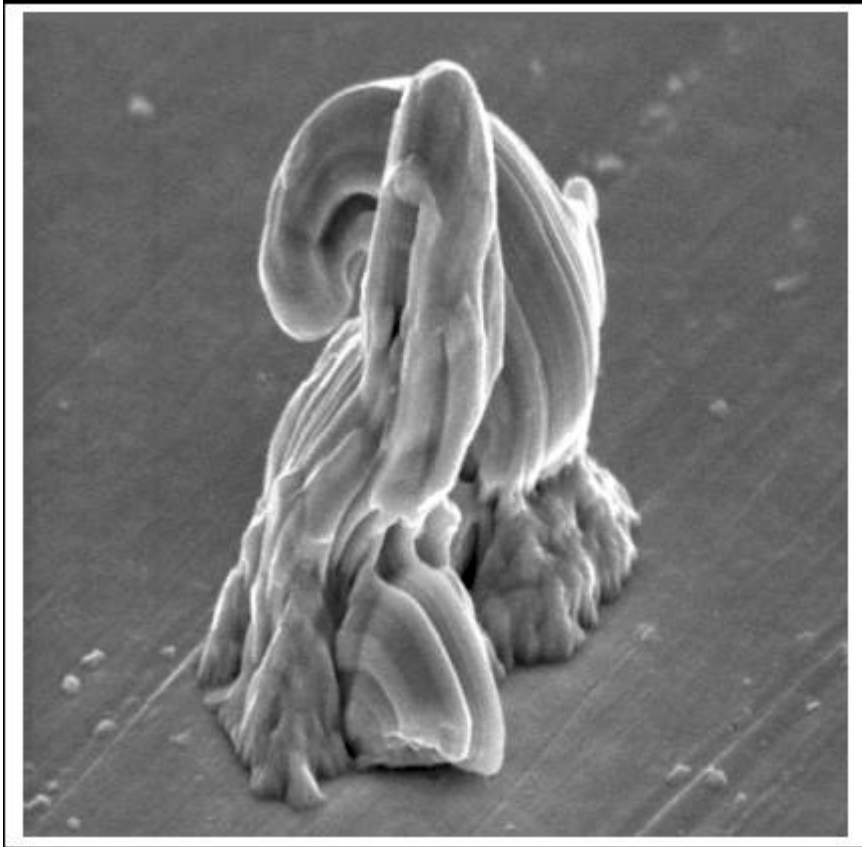


Figure 11

Magnification = 6600X

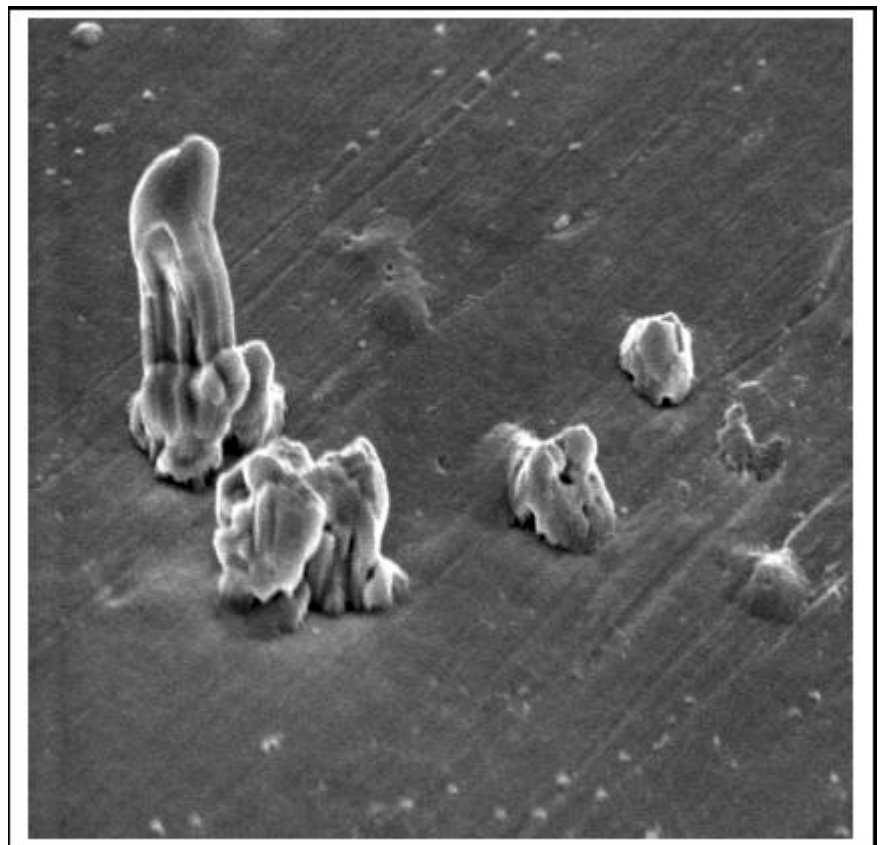


Figure 12

Magnification = 3800X

Reference:

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