

Solder Joint Embrittlement Mechanisms, Solutions and Standards

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Abstract

The change to lead-free solders in electronic assemblies created a need to replace tin-lead solderable termination finishes with materials such as pure tin or soft gold, on electronic components and substrates. Gold presented a risk of solder joint embrittlement, which could reduce the joint mechanical durability.

Several case studies were run, because solder joint embrittlement prevention requires a clear understanding of the materials and mechanisms of embrittlement.

We confirmed two known mechanisms and verified two other ways in which gold finishes can degrade solder joints. The known mechanisms are, 1) A gold layer dissolves from one side of a surface mount joint and precipitates AuSn_4 compound onto the opposing termination and 2) The gold fully dissolves from a surface mount termination, but it results in an excessive gold weight percentage in the joint. The other ways are, 3) A manual soldering process temperature is high enough to dissolve some nickel along with the gold and 4) A slow dissolving, hard gold surface finish incompletely dissolves during plated-through-hole soldering and solid state diffusion forms an AuSn_2 compound layer.

Close-up and cross-sectional images with SEM/EDS compositional information are shown for each case. A table of solder and gold volumes, which produce 3.0 and 4.0 weight percent gold, is provided. The embrittlement problems and their reliability solutions are discussed (over twenty literature references).

The data suggests how to improve gold plating requirements, for solder joint embrittlement prevention, in solder assembly industry standards.

Introduction

The manufacture of reliable radar electronics hardware relies on high quality metallic finishes on electronic components and interconnecting substrates. The assembly manufacturing producibility parameters which motivate high finish quality are solderability and wire bondability.

Gold and palladium are widely used in the finish designs, in order to enhance solderability and wire bondability. A thick, pure gold finish can be used to enhance wire bondability. However gold sufficiently thick for wire bonding can be too thick for soldering, causing solder joint embrittlement. Also, a component might require gold as a mating or wear surface, resulting in a gold design which is too thick and/or too impure for optimal soldering.

If the gold or palladium dissolution is excessive during the solder alloy's liquid phase formation of a solder joint, then the composition, mechanical properties and durability of the resulting joint alloy can change, compared with the original solder alloy.

Electronic assemblies are designed to work functionally with a given solder alloy. A change of composition will influence mechanical properties and the resulting durability can be unknown or degraded.

Degradation can occur in the as-built condition or after exposure to the environmental stresses of the application. If the gold or palladium is incompletely dissolved during soldering, then solid state diffusion can occur between the residual gold or palladium finish and the solder in the joint, causing a reliability concern from a metallurgical change during the use life of the hardware.

Given the above understandings, solder joint embrittlement is defined as a change of solder joint durability, due to dissolution and/or reaction with a finish such as gold and/or palladium.

The changes are expressed in tin-based solders by the appearance of AuSn_4 or PdSn_4 intermetallic compounds, from gold and palladium finishes, respectively. The compounds can occur in the bulk of the solder joint, at the finish interface or in both locations. The compounds are brittle in comparison with the soft solder alloy. As a result, the ability of the joint to be robust when subjected to mechanical strains is reduced.

Basic mechanical properties such as impact strength and strain rate sensitivity can change, depending on the composition of the solder joint, affected by the amount of compound added during solder joint formation.

If gold-tin or palladium-tin compounds other than the most tin-rich ones mentioned above were present in the tin-rich solder joint, then it would indicate an equilibrium condition was not achieved during solder joint formation. As a result, the metallurgical reliability of the joint would be further suspect. The joint properties would change as the joint attempted to reach equilibrium, during the electronic hardware's use life. (The metallurgical rate of change is a function of absolute temperature.)

Problem Statement and Methodology

The challenge to successfully use gold finishes increased when the tin-lead finishes were banned from low criticality applications like phones and cameras, due to a concern about leaching of lead (Pb) from waste electronics into water supplies. (The concerned seek environmental health peer review of low-lead, whisker preventing finish solutions like Sn90Pb10.)

As a result of the ban, the use of gold and pure tin increased. However, the pure tin finish had a shorting and fusing issue from growing whisker-shaped filaments¹, when plated without the whisker ameliorating element, lead.

The challenge for gold is that it, as well as palladium^{2,3,4} and copper⁵, can cause embrittlement of soft solders. A maximum gold thickness needs to be known, to prevent excessive embrittlement.

Electrolytic gold has a need for a minimum thickness as well, in order to prevent porosity, which can reduce solderability quality. Please refer to Figure 1, describing a hot cyanide gold plating process.

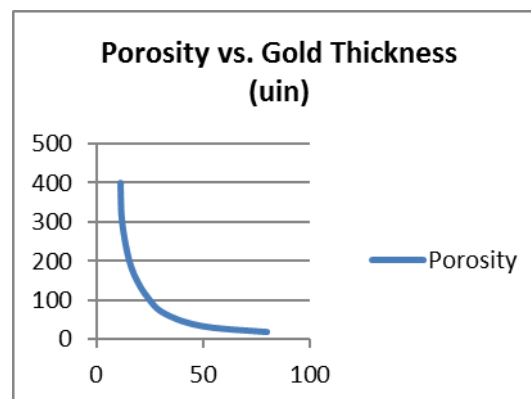


Figure 1. Porosity versus Electrolytic Gold Thickness from 11.4 to 80 Microinches Thick (after Krumbein⁶).

However, gold generally is regarded as a highly solderable finish^{7,8,9}.

The present work gives case study examples of various solder joint embrittlement material mechanisms, each with its manufacturing or design solution. The solutions are based on existing industry standards and other reported work.

After reviewing and summarizing weight percent calculations, by inputting typical gold and palladium plating and solder volumes into a weight percent calculator, some opportunities for standards improvements are suggested.

The goals are solder joint embrittlement prevention and an easy reliance on gold and palladium finishes for soldering and wire bonding.

Case Study 1 – Gold-Tin Compound Precipitates Onto an Interface

Case Study 1 Data

A connector pin with gold over nickel finish design was hot solder dipped with Sn63Pb37 solder alloy. The gold layer was fully dissolved. Gold dissolves very quickly¹⁰ in molten tin-lead solder.

The pin then was soldered on its side to a circuit board pad, also having gold over nickel finish design. The pad was not hot solder dipped, because its gold was deemed thin enough to avoid excessive gold embrittlement in the solder joint.

After soldering, a solder joint crack was evident at the location of the pin, as shown in Figure 2.

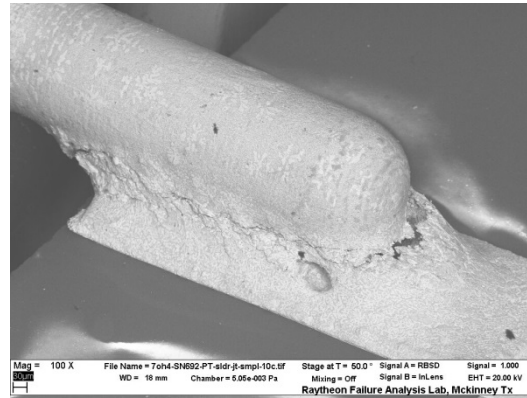


Figure 2. Solder Joint from Pin to Surface-Mount Circuit Board Pad Exhibited a Crack (100X).

The joint was submitted to failure analysis. Cross-sectional data was obtained as shown in Figure 3.

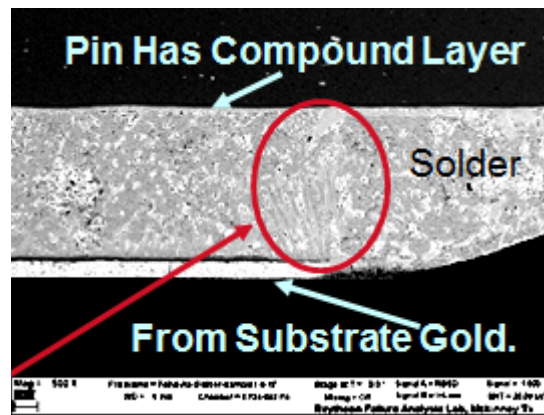


Figure 3. Gold dissolved from Board Pad on the Substrate (Bottom Black Layer), and Was Evident as Compound in the Joint (See Oval) and on the Pin (Top Black Layer) (500X).

A board pad was examined and found to have a perimeter region, which lacked the plated nickel diffusion boundary layer, as shown in Figure 4. To be clear, the nickel design was meant to cover a thick gold layer completely, and have its own overcoat of thin, solderable gold.

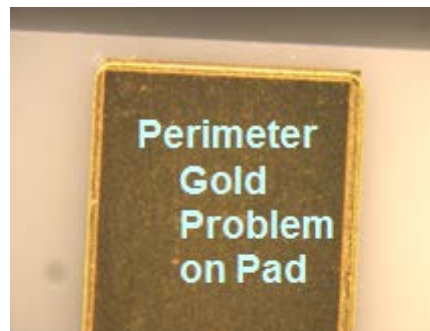


Figure 4. Top View of Circuit Board Pad Shows Nickel Did Not Cover Thick Gold's Perimeter.

Case Study 1 Results

The substrate pad design had gold over nickel, but the nickel area did not quite cover all of a layer of gold under the nickel. A perimeter region of thick gold had access into the solder joint.

During the soldering, the perimeter gold dissolved profusely. Upon solidification, much of the gold-tin compound actually precipitated onto the pin on the other side of the joint. As the joint began to cool and solidify, first the AuSn₄ precipitated (217 °C solidification temperature) onto the pin, and in the joint. Then tin-lead solder (183 °C solidification temperature) solidified onto the AuSn₄, leading to a weak interface at the pin.

The connector pin had been hot solder dipped, but the gold-tin compound precipitated onto it, causing a weak joint, which cracked at the pin.

The image in Figure 5 shows how our understanding was applied. We covered the gold pad completely with an overlay of nickel.

The result was a strong joint, free of embrittlement.

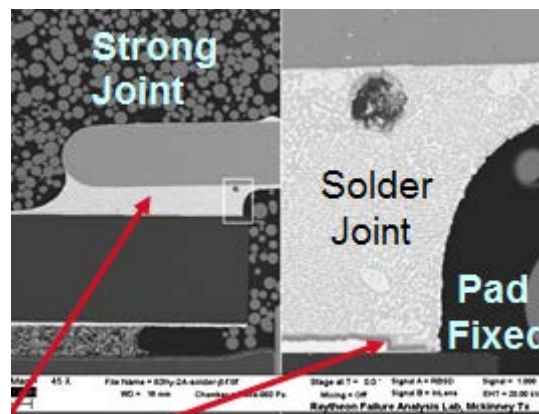


Figure 5. Cross-Section of Improved Pad Plating Design (45X), with Close Up View on the Right (600X).

As indicated with the arrow on the right in Figure 5, when the thin, dark grey layer of nickel completely covered the top and the side of the thick gold perimeter, the result was no evidence of gold-tin intermetallic compound in the solder joint. Bester¹¹ observed a 1-2 weight percent gold content is needed before the acicular or platelet structure of the gold-tin compound can be observed in Sn63Pb37 solder alloy

The solder interface separation at the pin was solved by an improved plating design on the pad. There was no profuse AuSn₄ formation from the pad, no detectable AuSn₄ on the pin or in the joint, and most importantly, no weak interface at the pin.

Case Study 2 – Excessive Gold in Soft Solder Joint

Case Study 2 Data

A surface mount soldered connector pin did not always survive mating and demating. The connector is shown in Figure 6 and the pin joint failure is shown in Figure 7.



Figure 6. Surface Mount Connector Center Pin Sometimes Failed Mate/Demate.

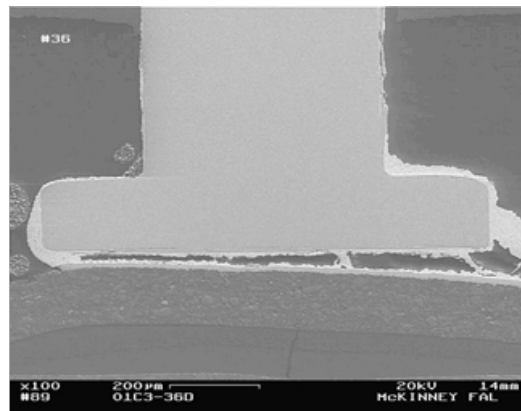


Figure 7. Cross-Sectional Image of Failed Pin Solder Joint (100X).

A close up view of the fillet on the left of Figure 8 is shown in Figure 9. A similar close up fillet view in Figure 10 was measured for composition using Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM/EDS). The volume of inspection included most of the solder joint area. The result was a 10 weight percent gold content. The solder alloy was SnAg3.7. A second solder joint area similarly was measured and found to have an 11 weight percent gold content.

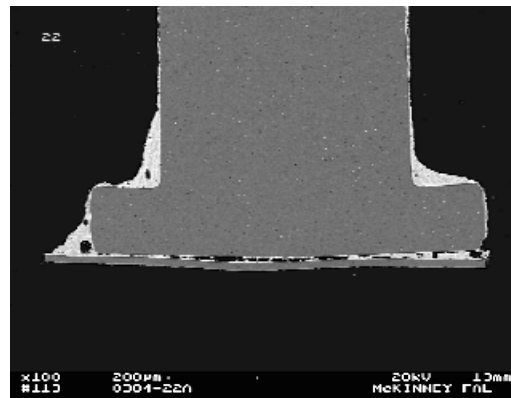


Figure 8. Cross-Sectional Image of Pin Solder Joint (100X).

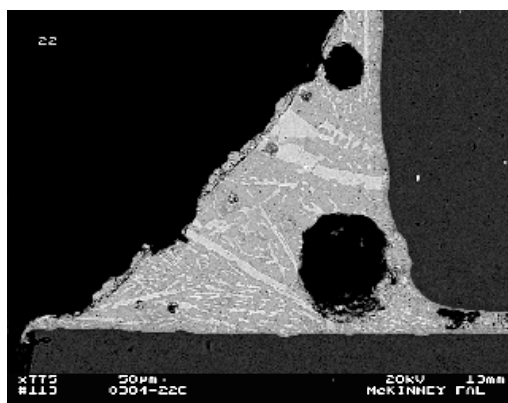


Figure 9. Close Up Image (775X) of Left Solder Fillet in Figure 8.

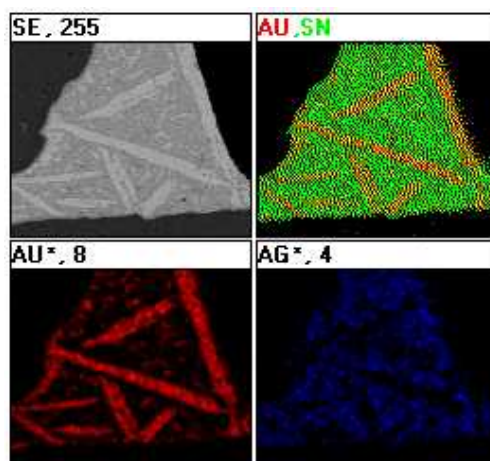


Figure 10. Fillet Similar to Figure 9, Showing Gold (Au), Tin (Sn) and Silver (Ag) Morphology.

A 32 run experiment varied solder joint preform quantity, wicking procedure and three solder process temperature factors. The experiment was specific to the unique equipment and process used in pin solder joint assembly.

Case Study 2 Results

The optimal levels for each factor had the result of increasing the pin joint pull strength significantly, shown in Figure 11, and demonstrated in Figures 12 -14.

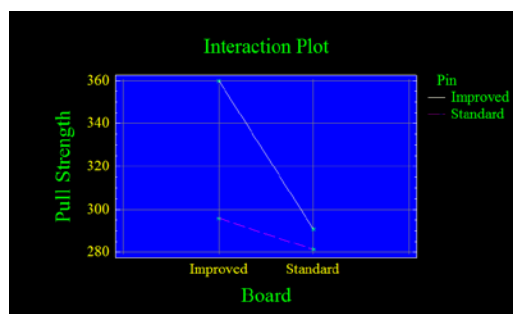


Figure 11. Reducing the Gold on the Pin and Board, and Increasing the Solder Volume on the Board, Interacted Beneficially to Increase Pin Pull Strength (grams).



Figure 12. During the Pin Pull Test, the Pin-to-Trace Solder Joint was So Strong that the Trace Pad and the Board Material were Torn Out of the Board.

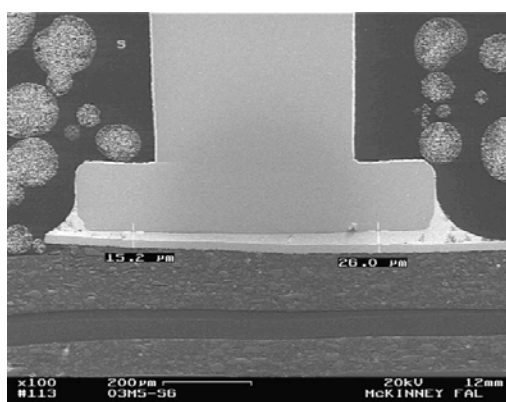


Figure 13. Solder Joint Quality Improvement Was Verified in Terms of Reduced Gold Content And Reduced Voiding (100X).

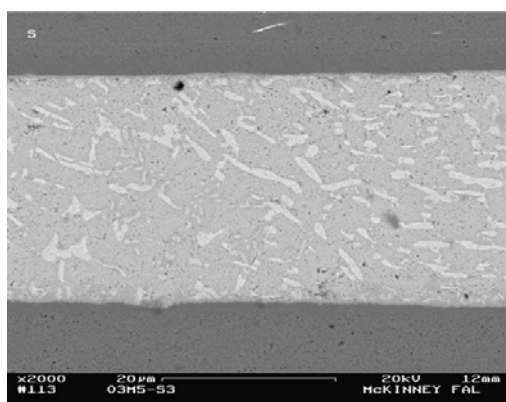


Figure 14. Close Up View (2000X) of the Solder Joint in Figure 13 Demonstrates the Elimination of Residual, Undissolved Gold Layers on the Pad and the Pin with the Experimental Design's Optimal Settings.

The excessive gold in the soft solder joint was eliminated by running an experimental design including pre-assembly gold removal steps, per a designed experiment with a validation run.

Case Study 3 – Manual Soldering with High Temperature

Case Study 3 Data

A failure analysis of the soldered surface mount connector shown in Figure 15 revealed solder joint cracking was associated with excessive gold and nickel in the Sn63Pb37 solder alloy. The horizontal crack shown in the foreground of the figure was investigated with cross-sectional analysis, per Figures 16. Metallurgical phases in the microstructure of the cross-section were evaluated for their compositions with SEM/EDS.

Table 1 shows the overall composition of the solder joint, where the area of inspection included the overall joint in Figure 16. The area did not include the interface region, Spectrum 1, to avoid error from nearby nickel signal. Four (4) measurements were made on different cross-sections, providing the following result. The gold average was 3.6 weight percent. The nickel average was 1.3 weight percent.

Table 2 shows the composition of the medium grey phase in Figure 16, marked as Spectrum 2. Per the atomic percent data, the intermetallic compound phase is identified as $(\text{Au}_{0.45}\text{Ni}_{0.55})\text{Sn}_4$.

Spectrums 3 and 4 in Figure 16 were identified as the tin-rich and lead-rich phases, respectively.

Additional data was obtained on the as-made gold thickness of the connector. Its gold thickness was 0.70 microns (28 microinches). The circuit board had an electroless nickel immersion gold surface finish.

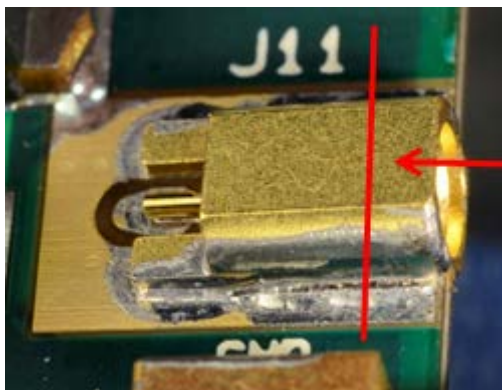


Figure 15. Soldered Surface Mount Connector with Joint Crack. Arrow Points to the Sectioning Plane.

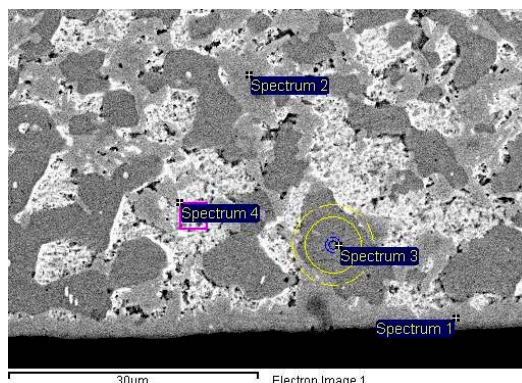


Figure 16. Section of the Cracked Joint, with Close Up of Solder Attached to the Circuit Board Pad, (2200X).

Table 1. Composition of the Overall Solder Joint in Figure 16.

Element	Weight%	Atomic%
Ni K	1.24	2.85
Sn L	66.71	76.08
Au M	3.82	2.62
Pb M	28.23	18.45
Totals	100.00	100.00

Table 2. Composition of the Phase Marked as Spectrum 2 in Figure 16.

Element	Weight%	Atomic%
Ni K	5.39	10.93
Sn L	79.91	80.18
Au M	14.71	8.89
Totals	100.0	100.0

Case Study 3 Results

A multi-functional team reviewed possible failure causes in terms of the following categories.

1. Materials
2. Machine
3. Process
4. Environment
5. People
6. Method

The materials category had two causes. The process and method categories each had one cause.

The two causes in the material category were the gold thickness and the flux used. The gold thickness resulted in a gold weight percent at the maximum 3 to 4 weight percent gold limit in the IPC-AJ-820A specification (pp. 39, 156-157). The flux required excessive time at temperature during manual soldering.

The process category cause was a lower than desired yield, leading to higher than desired rework.

The method category cause was a soldering setup with low heat sinking, which motivated a higher manual soldering temperature. The high manual soldering temperature was deduced from the nickel content in the identified phase, $(\text{Au}_{0.45}\text{Ni}_{0.55})\text{Sn}_4$, whose morphology was anomalous compared with the typical acicular or platelet structure of the expected compound, AuSn_4 .

The manual soldering with high temperature was corrected by four changes. These were, 1) a thinner gold finish on the surface mount connector, 2) a change to a more producible flux for manual soldering, 3) an improved first pass soldering process with higher yields, and 4) an improved rework process with a setup providing more heat sinking.

The present analysis of manual soldering with high temperature demonstrates that the high temperature can cause significant nickel dissolution in addition to the gold dissolution. Unlike the case of embrittlement from gold-tin compound, there is not an industry accepted and specified weight percent limit for embrittlement with gold-nickel-tin compound.

Case Study 4 – Hard Gold Surface Finish

Case Study 4 Data

A pin with a hard gold over nickel finish was soldered to a circuit board's plated-through-hole, in a configuration as shown in Figure 17. The resultant joint, made with Sn63Pb37 alloy, was found to have a fillet crack after soldering, as shown in Figure 18.

Cross-sectional analysis revealed that in the fillet area, not all of the gold was dissolved from the pin during soldering.

An as-like solder joint was examined more rigorously. Its crack was found to be located specifically between a residual gold layer on the pin and an AuSn_2 intermetallic compound layer, as shown in Figure 19. Adjacent to the AuSn_2 was a contiguous AuSn_4 layer. In addition, typical AuSn_4 compounds were found to be distributed throughout the solder fillet.

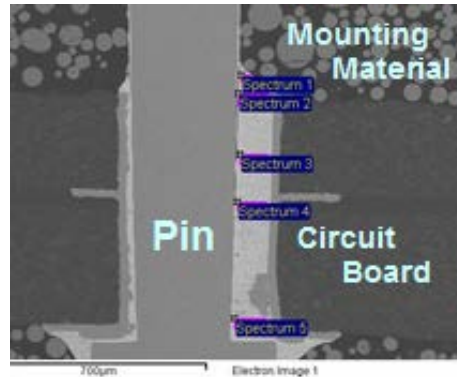


Figure 17. Cross-Sectional View of Pin in Plated Through Hole Solder Joint (150X).

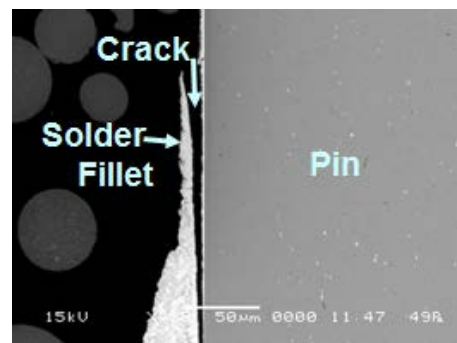


Figure 18. Close Up View of a Solder Fillet Crack Showing Separation from the Pin (275X)

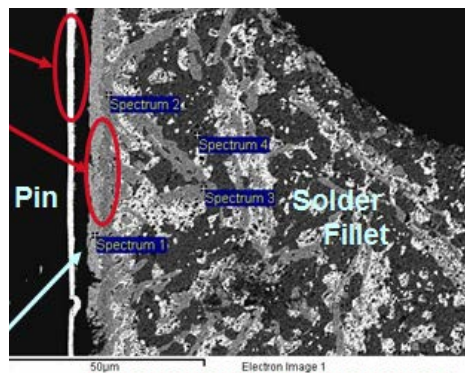


Figure 19. Microstructure of a Crack, Showing Residual Gold Layer (Top Oval), Crack (Lowest Arrow) and Duplex $\text{AuSn}_2/\text{AuSn}_4$ Compound Layer (Other Oval) in the Fillet (700X).

It is useful to refer to the melting points, which are shown in Table 3. The melting point of the AuSn_2 was not reached during soldering. It is proposed that the high melting point AuSn_2 compound formed by solid state diffusion. In contrast, gold dissolution and reaction with tin formed the AuSn_4 while the solder was still molten.

Accordingly, fast diffusion¹² between the gold and the AuSn_4 occurred during cooling of the solidified solder joint. The diffusion formed the adjacent AuSn_2 layer.

Note that the AuSn_2 occurred only as part of the duplex $\text{AuSn}_2/\text{AuSn}_4$ compound interface layer. The AuSn_2 was not distributed in the solder fillet.

The diffusion weakened the gold-to-AuSn₂ interface, and the localized fillet contraction produced a peeling stress sufficient to cause the crack. Given that plated layers may contain up to twenty percent vacancies¹³, the solid state diffusion can result in an accumulated vacancy interface, thereby creating a weakened or voided layer.

In addition, solid state diffusion and reaction rates are known to depend upon the surface finish's state of stress¹⁴, produced by the plating process.

Table 3. Melting Points of Solder and Compounds (°C)

Sn63Pb37 Solder Alloy	183
AuSn ₄ Gold-Tin Compound	252
AuSn ₂ Gold-Tin Compound	309

Case Study 4 Results

The analysis determined that the AuSn₄ formed during soldering and was attached to the residual gold layer. During the cool down, just after solder joint solidification, an additional intermetallic compound, AuSn₂, formed at the interface and caused the crack.

It is possible that the hard gold finish was causal to the AuSn₂ formation (and hence to the crack), not just because the finish was gold, but also because it contained a nickel or similar hardening element.

Hard gold can have at least three to ten times the impurity level as soft gold, per Table 4. The hardening impurities can have a dissolution rate in solder, which is much slower than gold's dissolution rate.

Nickel dissolves in a 230C, molten Sn60Pb40 solder bath at the rate of 0.05 microinches per second, and gold at 100 microinches per second, per Figure 20.

The hardening of the gold slowed the gold dissolution rate, resulted in more/residual gold thickness after solder joint solidification, increased the amount of diffusion and AuSn₂ formation, and was causal to the cracking.

Table 4. Gold Hardness and Purity Specifications

Gold Hardness	Gold Specification (Type describes Purity; Code describes Hardness)	Minimum Mass Percent Gold
Soft	ASTM B488-11, Type III, Code A	99.9
Hard	ASTM B488-11, Type II, Codes B, C & D	99.0
Soft & Hard	ASTM B488-11, Type I, Codes A, B & C	99.7

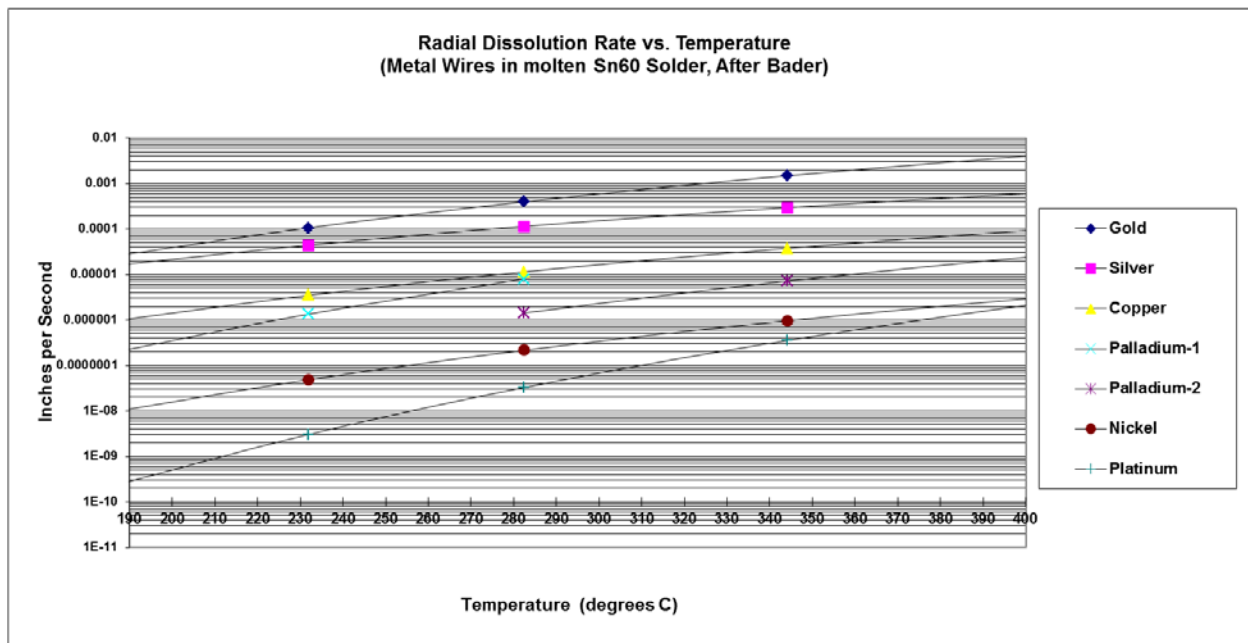


Figure 20. Radial Dissolution Rate vs. Temperature of Metal Wires in Molten Sn60Pb40 Alloy, After Bader¹⁰ (Chart Extrapolates Beyond Data Ranges).

Regarding the hard gold surface finish, the solution was to hot solder dip the pin in a controlled manner to fully dissolve the gold thickness. As a result, the pin had a solderable finish, which was virtually free of gold. Thereby the embrittlement problem was solved.

A lesson learned is that it is important to cross-section and check to ensure there is complete gold dissolution during formation of the solder joint.

If the hardness and purity of the gold are not known, then the cross-section becomes all the more important to validate complete gold dissolution, and thereby to ensure prevention of solder joint embrittlement.

Suggestions for Standards

Standards – Establishing Gold Weight Percent, Palladium Weight Percent and a Calculator Equation

The IPC-AJ-820A Assembly and Joining Handbook¹⁵ calls out a 3 to 4 weight percent maximum gold limit in a solder joint. The limit is consistent with referenced proposed limits^{16, 17, 18}, ranging from 3 to 5 weight percent.

Wild¹⁹ reported that up to 1.0 weight percent gold actually increases the strength of the Sn63Pb37 alloy; and the solder's ductility and toughness are improved with up to 2.5 weight percent gold.

Bend tests show 5.0 weight percent gold in tin is mechanically acceptable²⁰.

Vianco² provided guidance that the maximum palladium limit in a solder joint should not exceed 2 weight percent.

A simple equation²¹ can be used to create a solder joint embrittlement prevention (SJEP) spreadsheet calculator. A simplified version of the equation is shown in Figure 21.

	Wt%(Au)	=	$\frac{100\% \times [V(\text{Au}) \times D(\text{Au})]}{[V(\text{Au}) \times D(\text{Au})] + [V(\text{Sn63}) \times D(\text{Sn63})]}$
where			
	V(Au)	=	Volume of gold
	V(Sn63)	=	Volume of solder joint
	D(Au)	=	Density of gold
	D(Sn63)	=	Density of Sn63 solder
	Wt%(Au)	=	Weight percent gold in the solder joint

Figure 21. Equation for Calculating the Weight Percent of Gold in a Soft Solder Joint

Standards – Deriving Maximum Gold and Palladium Thicknesses for SJEP

Solder joint embrittlement prevention (SJEP) makes use of the equation in Figure 21. The equation easily can be modified for palladium, using palladium’s density to calculate the weight percent of palladium in a soft solder joint.

Individual calculations were made, for each of the entries shown in Figure 22. The solder alloy was Sn63Pb37. A simple solder joint design was used, where the area of the solder joint equaled the area of the finish, for example, 100 mils by 100 mils.

Please note that the solder joint thickness was taken to be the solidified solder thickness (not the solder paste thickness). For example, a 3.0 mil solder joint thickness in Figure 22 would have been assembled with a solder paste stencil of about 6.0 mils thick.

In Figure 22, the four bolded rectangles show the gold or palladium weight percentages where the maximum allowable weight percent was nearly reached. The maximum for gold was set at 4.0 weight percent. The maximum for palladium was set at 2.0 weight percent.

Here is the result of Figure 22. There is a trend, where 14.2 microinch palladium thickness is equivalent to 18.0 microinch gold thickness, in terms of solder joint embrittlement prevention. Very thin plating is within the set weight percent limits for SJEP when the following conditions apply:

Gold 0.45 μm [18 microinches] or less on each side of an “equal area” solder joint, i.e., on both a surface mount component and a circuit board, or palladium 0.35 μm [14 microinches] or less on each side of an “equal area” solder joint, are acceptable when the gold is soft and pure per ASTM B488-11, Type III, Code A²², and when the solder joint is at least 2.0 mils thick (equivalent to a 4 mil thick stencil).

When the gold or palladium plating is only on one side of an “equal area” solder joint, the above plating, per the weight percent settings, is thin enough to avoid embrittlement if the joint is at least 1.0 mil thick.

1. Thin Gold and Palladium Calculations												
1.1 Weight Percent Gold												
Solder Joint Thickness (mils)	Microinches Gold on Component with zero			Microinches Gold on Substrate			Microinches Gold on Component with same			Microinches Gold on Substrate		
	8	15	18	0	0	0	8	15	18	8	15	18
1.0	1.78	3.29	3.93				3.51	6.38	7.56			
2.0	0.90	1.67	2.00				1.78	3.29	3.93			
3.0	0.60	1.12	1.34				1.20	2.22	2.65			
4.0	0.45	0.84	1.01				0.90	1.67	2.00			
1.2 Weight Percent Palladium set at 50% of the Weight Percent Gold												
Solder Joint Thickness (mils)	Microinch Palladium on Component with 0			Microinches Pd on Substrate			Microinches Pd on Component with same			Microinches Palladium on Substrate		
	6.4	11.9	14.2	0	0	0	6.3	11.7	13.9	6.3	11.7	13.9
1.0	0.89	1.65	1.97				1.75	3.19	3.78			
2.0	0.45	0.84	0.99				0.88	1.62	1.93			
3.0	0.30	0.56	0.67				0.59	1.09	1.29			
4.0	0.22	0.42	0.50				0.44	0.82	0.97			

Figure 22. Calculated Weight Percent of Gold and Palladium for Selected Solder and Finish Designs

Table 5. Example of How to Specify Electrolytic Gold Thickness Range for SJEP and Solderability

Maximum Gold Weight Percent in Solder Joint	Solidified Solder Thickness (mils)	Minimum Gold Thickness for Solderability (microinches)	Calculated Maximum Gold Thickness for SJEP
4.0	3.0	20	54
3.0	3.0	20	40
4.0	2.0	20	36
3.0	2.0	20	27

Standards – Considering the Possibility of a Gold Thickness Range for SJEP and Solderability

Referring back to Figure 1, there is a tradeoff between the need for a minimum amount of electrolytic gold, to ensure solderability, and a maximum amount of gold, to ensure solder joint embrittlement prevention (SJEP).

Solderability might be considered acceptable for very thin (about 2 microinches) immersion gold. In contrast, for the case of electrolytic gold, a relatively thicker gold might be considered to be necessary to prevent porosity and to ensure solderability.

From Figure 1, one might expect a minimum of 20 microinches of gold would be necessary to ensure solderability. However, a porosity test is more severe than a solderability test. In addition, the knee in the porosity curve is expected to be process dependent, such that an optimized electrolytic gold process might be solderable with a thinner gold than a non-optimized one.

Table 5 shows an example of a gold thickness range which might be considered acceptable. In the example, 20 microinches might be used as the minimum electrolytic gold thickness for solderability assurance in a manufacturing environment, where solderability needs to be acceptable after a shelf life. Also in the example, 4.0 weight percent gold might be used as the maximum for SJEP. If gold were present only on one side of a solder joint, the result would be as shown in Table 5.

Standards – Obtaining Maximum Weight Percent Specification Limits for Pb-Free Solder Alloys

The determination and use of a maximum weight percent limit is helpful to prevent solder joint embrittlement, not just for the Sn63Pb37 and SnAg3.7 solder alloys described in the case studies of the present paper.

Some of the embrittlement studies and criteria are starting to become explored for Pb-free solders. For example, the SnAg3.0Cu0.5 (formerly²³ SAC305) solder alloy was investigated²⁴. There were indications that a maximum of 5.0 Wt% (weight percent) gold would be acceptable for the alloy, if copper finish were in contact with the solder. However if there are nickel barriers on both the component and substrate sides of the solder joint, then the copper dissolution is impeded, the compound formation is changed, and the maximum gold is reduced to 3.0 Wt.%.

It will be helpful to determine and apply a maximum weight percent limit for each Pb-free (lead-free) solder alloy used in the electronics assembly industry, using properties such as impact strength vs. gold weight percent¹¹.

Standards – Additional Topics

A community of suppliers and users of plated components and substrates might wish to pursue some additional, related topics described next.

While the case studies of the present paper showed solder joint designs susceptible in their applications to solder joint embrittlement failures, there might be numerous solder joint designs which are reliable with more than a 4.0 weight percent limit (gold in Sn63Pb37). For example, specific low stress and low strain applications might not have a concern with embrittlement. However, the time and expense of running complicated reliability validations or mechanical property based models could be prohibitive and expensive. There is the additional concern that a model could be based on a questionable assumption.

The determination of a weight percent limit would normally be based on actual solder joint failure, as compared with a lower limit observed for a metallurgical precursor of failure.

In addition, it is possible that a calculation of weight percent could be in error. For example, the 90% metal weight of a solder paste should not be applied as if it were a paste volume, when in fact a 50% paste volume would be correct.

Standardization of the SEM/EDS weight percent measurement method is another opportunity. Such a method would be helpful in verifying a weight percent calculation.

Summary

The assertion in J-STD-001F²⁵, that gold embrittlement is considered a defect in soldered electrical and electronic assemblies, highlights the need for a clear understanding of the following:

1. Solder joint embrittlement mechanisms,
2. Proven solutions and
3. Specific standards opportunities.

The present paper describes four case studies of solder joint embrittlement, three with Sn63Pb37 and one with SnAg3.7 solder alloy. Solutions are given in each case. Applicability of the solutions to lead-free alloys and standards is addressed.

In the first case, a gold-tin compound was shown to precipitate onto a surface mount lead that had no gold on it. The substrate had a thick gold that was supposed to be capped with nickel, but the perimeter of the thick gold had not been capped, causing the gold to dissolve profusely during soldering. The case was illustrative of the common embrittlement mechanism of gold dissolution followed by AuSn₄ intermetallic compound formation and precipitation onto an interface, as well as into the solder joint bulk. A simple redesign of the nickel cap solved it.

In the second case, a surface mount soldered connector pin and the substrate pad both had too much gold, and the solder volume was too small, resulting in too high of a gold weight percent. We measured the gold content in the solder joint by Scanning Electron Microscopy with Energy Dispersive Spectroscopy, showing that one does not have to always rely on calculations. A 32 run experiment optimized the gold removal processes, the solder volume and three temperature settings for the unique manufacturing equipment. Validation cross-sections showed success.

While the first and second cases demonstrated commonly known mechanisms, the third and fourth cases provided some different information.

In the third case, manual soldering of a surface mount connector was performed. Solder joint cracking occurred during electrical test. A multifunctional team ran a cause-effect investigation, and found four causes. The manual soldering temperature was too high, found by microstructural analysis. Gold and nickel embrittlement resulted in (Au_{0.45} Ni_{0.55})Sn₄ intermetallic compound, with a morphology different from AuSn₄. We reduced the soldering temperature with a better heat sinking setup. Also, we improved the flux and used a connector with thinner gold.

In the fourth case, a hard gold surface finish on a pin caused a solder joint crack in a plated through hole application. Cross-sectional analysis revealed that in the fillet area, not all of the gold was dissolved from the pin. AuSn₄ at first was attached to the residual gold layer. AuSn₂ formed by diffusion, resulting in a duplex AuSn₂/AuSn₄ layer which broke from the gold. Gold hardeners have extremely slow dissolution rates in solder, compared with pure gold. The hardening of the gold slowed the gold dissolution rate, resulted in residual gold thickness after solder joint solidification, caused the AuSn₂ formation, and was causal to the cracking. Hard gold needs complete dissolution.

Regarding industry and standards opportunities, the case study findings and solutions are applicable to lead-free alloys.

We demonstrated how gold and palladium weight percent limits can be converted to maximum finish thicknesses, aiding solderability and wire bondability in assembly, as well as solder joint reliability in the field.

Based on their value to help prevent solder joint embrittlement, it would be helpful to determine the maximum weight percent limits for gold and palladium in each lead-free solder alloy used now, and in the future, by the electronics assembly industry.

Acknowledgements

The author gratefully acknowledges the company for permission to publish the paper, as well as the following sponsors, collaborators and analysts: Keith Kirchner, Bill Ortloff, Karl Worthen, John Bedinger, George Barson, David Pinsky, Anthony Rafanelli, Chris Bender, Jason Bain, Karla Willis, Emily Hoffman, Robert Champaign, Kennedy Coleman, Terry Banks, Shirley Smith, Dung Truong, Terry Graham, Marti Fountain, Theresa Hendricks, Michael Dwight and Marlin Downey.

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Solder Joint Embrittlement Mechanisms, Solutions and Standards

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Agenda

- Need to Reduce Risks from Lead-Free (Pb-Free) Finish Designs
 - Not Just Tin (whiskers) but also Gold and Palladium (embrittlement)
- Finish Quality Factors and Embrittlement Definition
 - Porosity, Plating Method, Solderability, Wire Bondability
 - Solder joint embrittlement is a decrease in solder joint durability, due to dissolution and/or reaction with a finish such as gold and/or palladium
- Four Case Study Examples of Embrittlement
 - Gold-Tin Compounds Precipitate onto an Interface
 - Excessive Gold in Soft Solder Joint
 - Manual Soldering with High Temperature
 - Hard Gold Surface Finish
- Standards: Weight %, Finish Thickness; Each Pb-Free Solder Alloy

Solder Joint Embrittlement Understanding Aids Reliable Solder Joint Design



Introduction

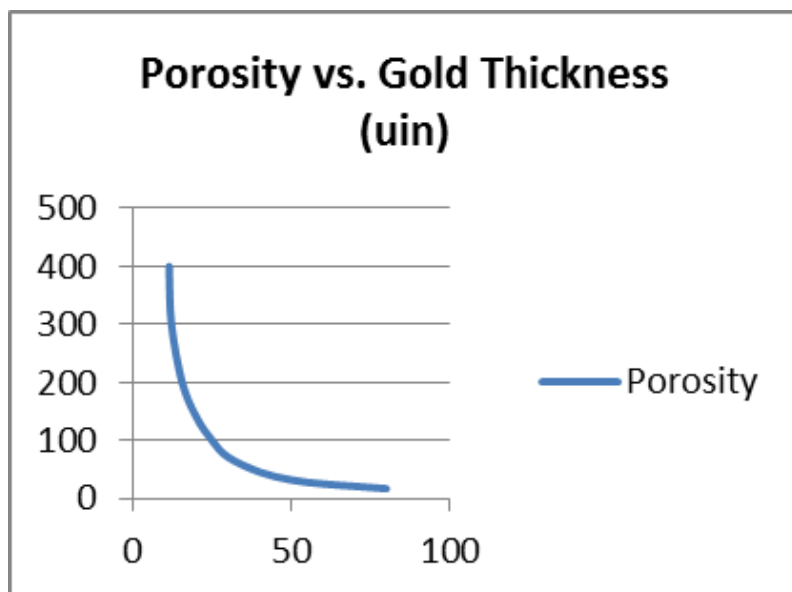
- Metallic Finishes on Electronic Components/Interconnecting Substrates
 - Gold Sufficiently Thick for Wire Bonding Too Thick for Soldering
 - Gold Design for Mating or Wear Too Thick or Too Impure for Optimal Soldering
- Solder Alloy Vs. Solder Joint Composition
 - Dissolution of Gold Influences Composition and Mechanical Properties
 - Durability Unknown or Degraded
- Soldering Process
 - Incomplete Gold or Palladium Dissolution Enables Solid State Diffusion
 - As-Built or During Application Environment
- Intermetallic Compounds from Gold and Palladium Finishes
 - AuSn_4 and PdSn_4 Hopefully
 - Acceptable and Unacceptable Levels
 - Brittle Compounds, Kirkendall Voiding In Joint Bulk and/or At Finish Interface

Soldering Finish, Pb-Free Alloy, Process & Compounds can Cause Embrittlement



Gold Thickness

- Gold Generally Regarded as a Highly Solderable Finish
- Maximum Gold Thickness Needs to be Known to Prevent Embrittlement
- Minimum Electrolytic Gold Thickness is Needed to Prevent Porosity



ASTM B735 Standard Test Method for
Porosity in Gold Coatings

Solderability a Function of Finish Process

- Immersion Gold (IPC 4552 & 4556)
- Electrolytic or Electroless Gold

**Figure 1. Porosity Versus Electrolytic Gold
Thickness from 11.4 to 80 Microinches
Thick (after Krumbein)**

Gold Design Thick Enough (as a Function of Finish Process) and Not Too Thick



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Gold-Tin Compound Precipitates onto Interface

Case Study 1 Data

- Connector Pin with Gold over Nickel Hot Solder Dipped, Gold Removed
- Sn63Pb37 Fully Dissolved Pin Gold; Side of Pin Soldered to Circuit Board

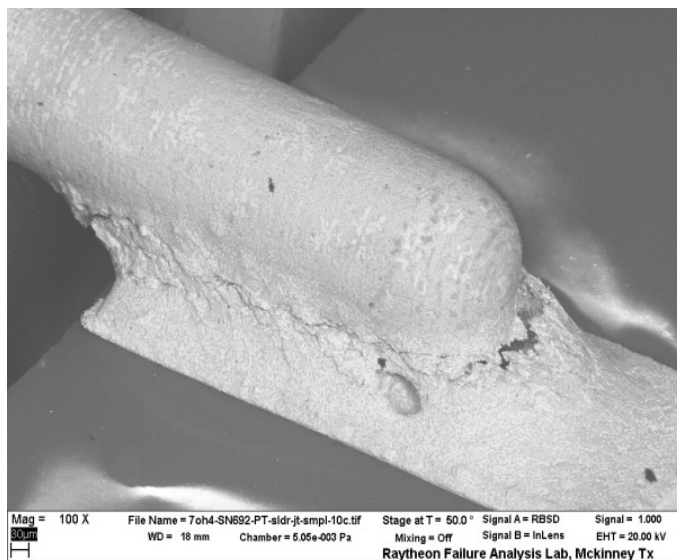


Figure 2. Solder Joint from Pin to Surface-Mount Circuit Board Pad Exhibited a Crack (100X).

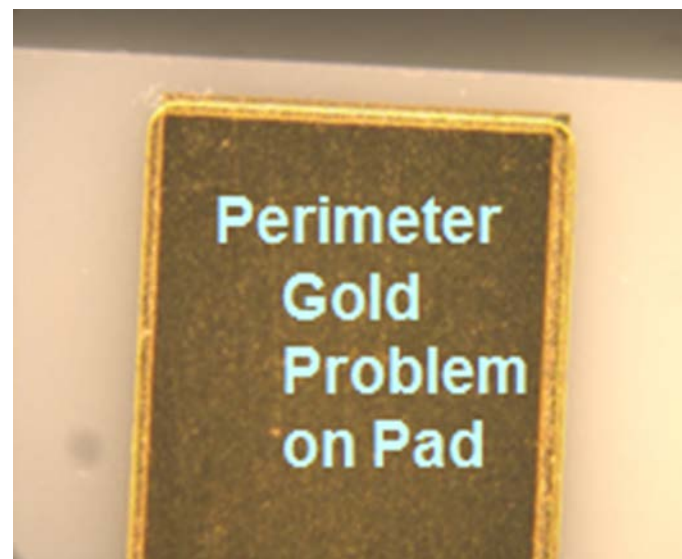


Figure 3. Top View of Circuit Board. Nickel Not Fully Covering a Thick Gold's Perimeter.

Avoid "Infinite" Source of Gold Available to Dissolve Into Solder Joint



Gold-Tin Compound Precipitates onto Interface

Case Study 1 Results

- The AuSn_4 compound precipitated into joint and onto pin. Then tin-lead solidified, leading to a weak interface at the pin.

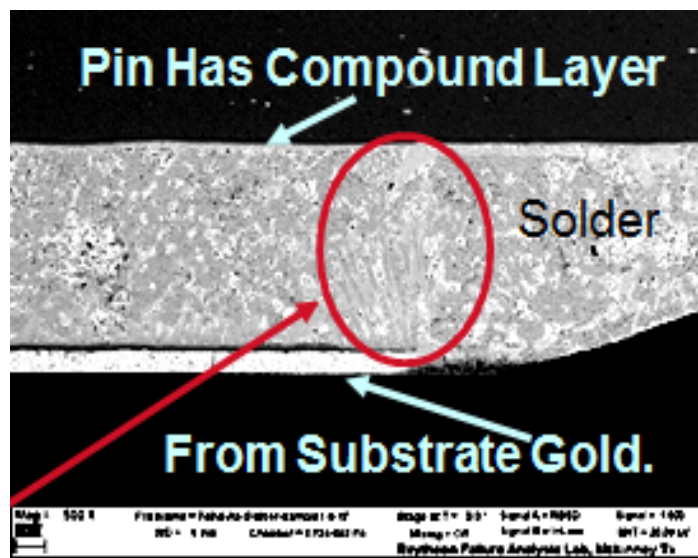


Figure 4. Gold dissolved from Board Pad on Substrate and Reacted into Compounds in Joint and on Pin (500X).

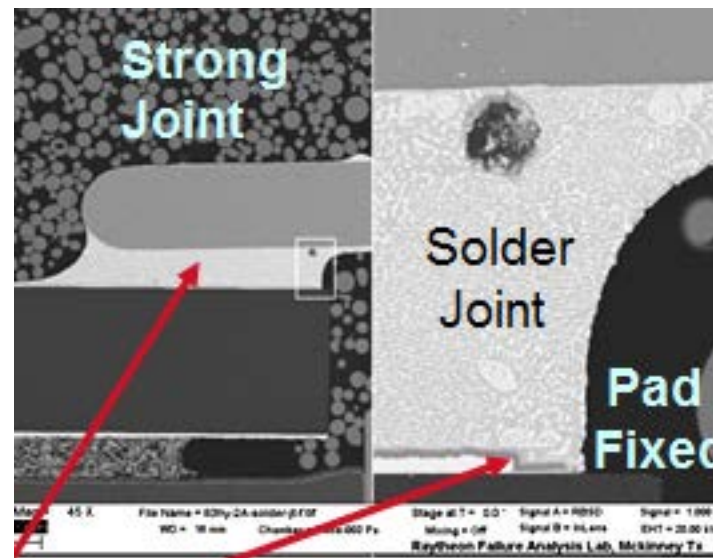


Figure 5. Cross-Section of Improved Pad Plating Design (45X), with Close Up View of Nickel on Right (600X).

Embrittlement Mechanism: Gold from Substrate Precipitated onto Pin



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Solder Joint Embrittlement Understanding Aids Reliable Solder Joint Design



Excessive Gold in Soft Solder Joint

Case Study 2 Data

- A surface mount soldered connector pin did not always survive mating and demating. The solder alloy was SnAg3.7.

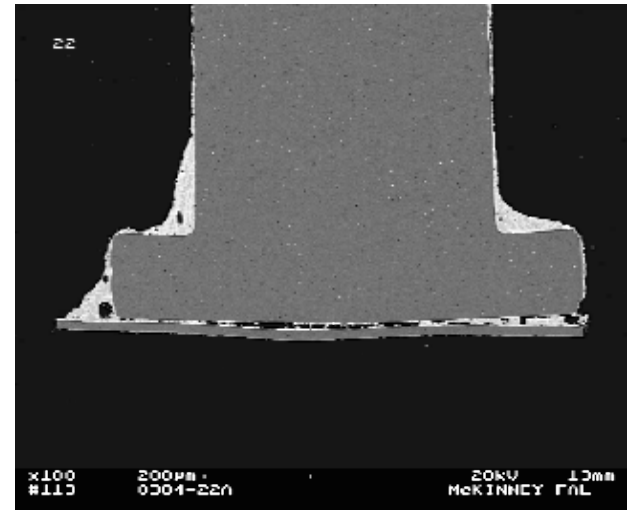
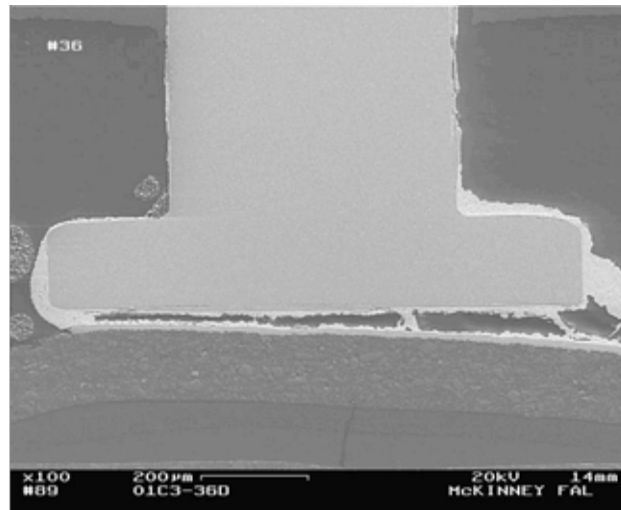


Figure 6. Surface Mount Connector's Center Pin Embrittlement Examination. Connector Top View (Left). 100X Cross-Sectional Views of Failed Pins (Right).

We Examined Gold Embrittlement of SnAg3.7 Soldered Connector Pin



Excessive Gold in Soft Solder Joint

Case Study 2 Results

- Solder Joint Composition Per SEM/EDS, Where Inspection Volume Included Most of the Fillet Area, Revealed 10-11 Wt% Gold.

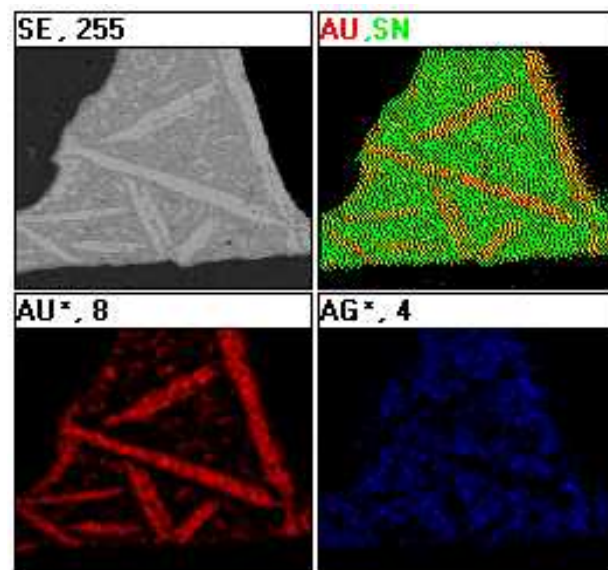
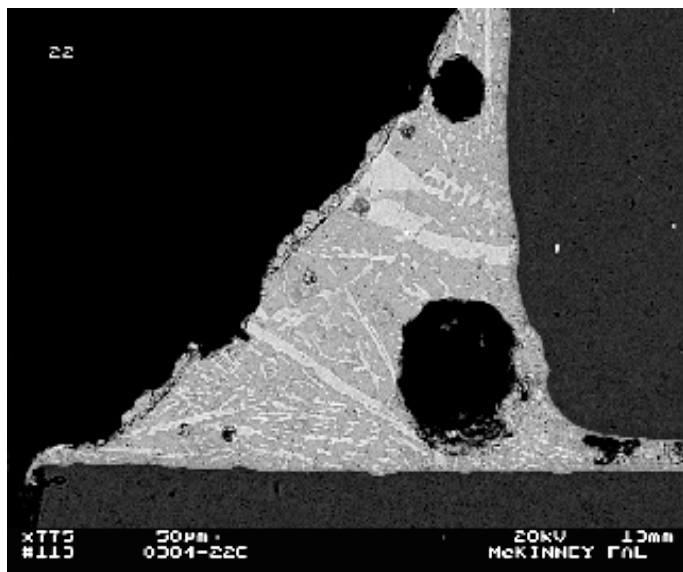


Figure 7. Close Up Image (775X) of a Solder Fillet from Prior Slide (Left). Digital Image of a Similar Solder Fillet Showing Gold (Au), Tin (Sn), (Ag) Morphology (Right).

10 Weight Percent Gold is Too High for SnAg3.7 Soldered Connector Pin



Excessive Gold in Soft Solder Joint

Case Study 2 Results

- 32 Run Experiment: Solder Preform Quantity, Wicking Procedure, Soldering Process.

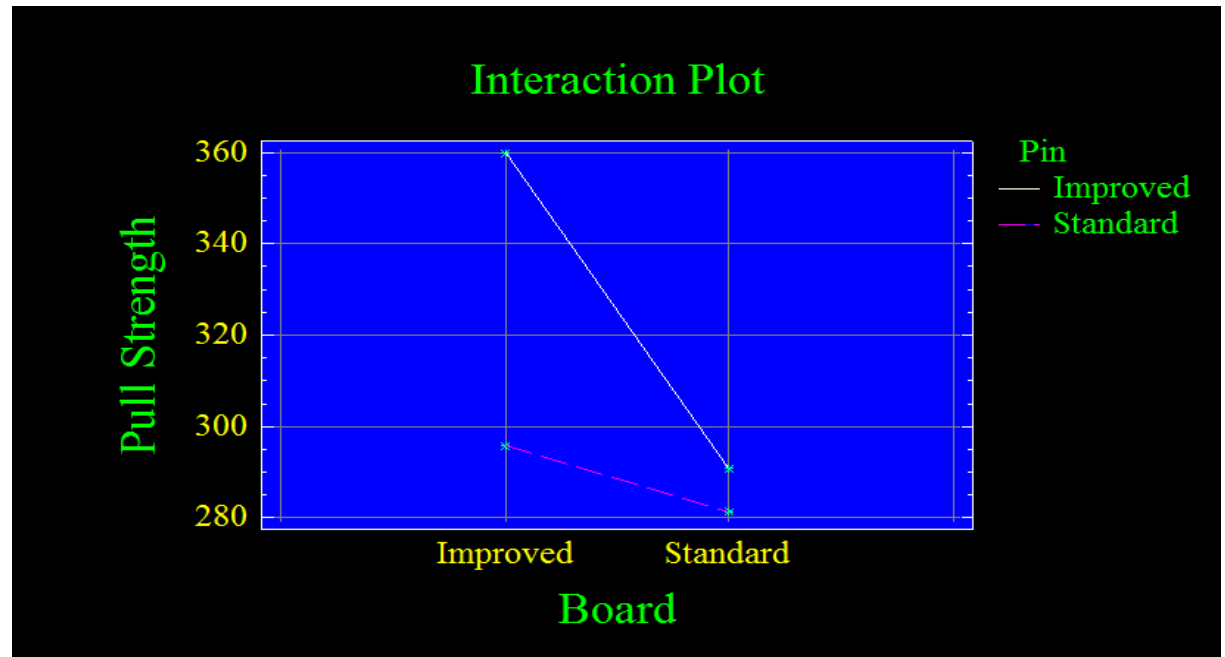


Figure 8. Optimal Levels for Each Factor Reduced the Pin and Board Gold, Increased the Solder Volume and Interacted Beneficially to Increase Pin Pull Strength (grams).

Optimized Parameters Interacted Per Experimental Design Interaction Plot



Excessive Gold in Soft Solder Joint

Case Study 2 Results

- Validation Run

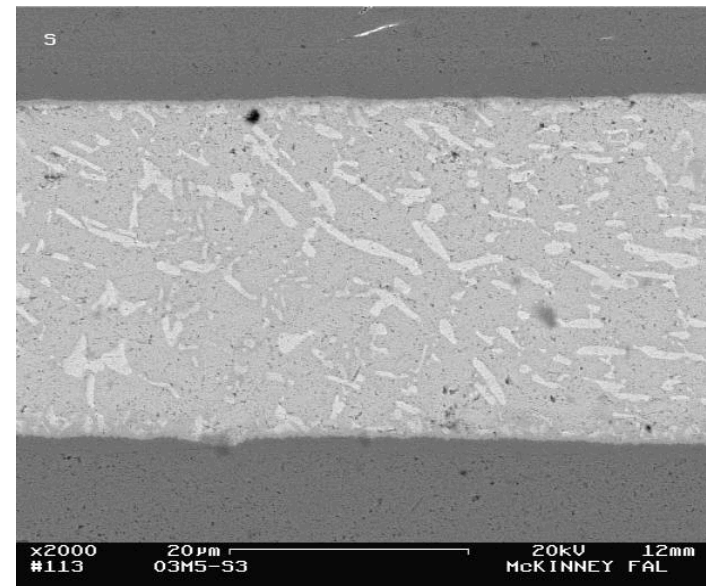
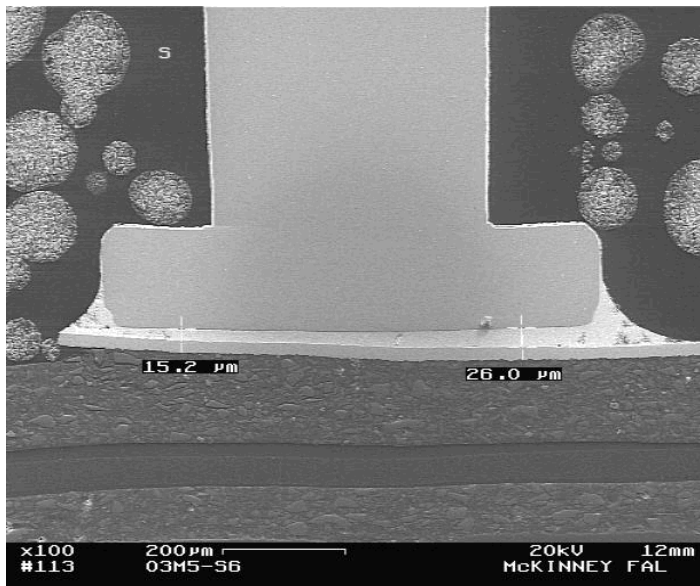


Figure 9. Cross-Sectional View of the Pin (100X) and a Close Up Solder Joint View (2000X) Show Joint Quality Improved, and Gold Content and Voiding were Reduced.

Solder Joint Embrittlement Can Be Prevented By Process and Design



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Solder Joint Embrittlement Understanding Aids Reliable Solder Joint Design



Manual Soldering with High Temperature

Case Study 3 Data

- Solder Joint Cracking was Associated with Excessive Gold and Nickel in the Sn63Pb37 Solder Alloy

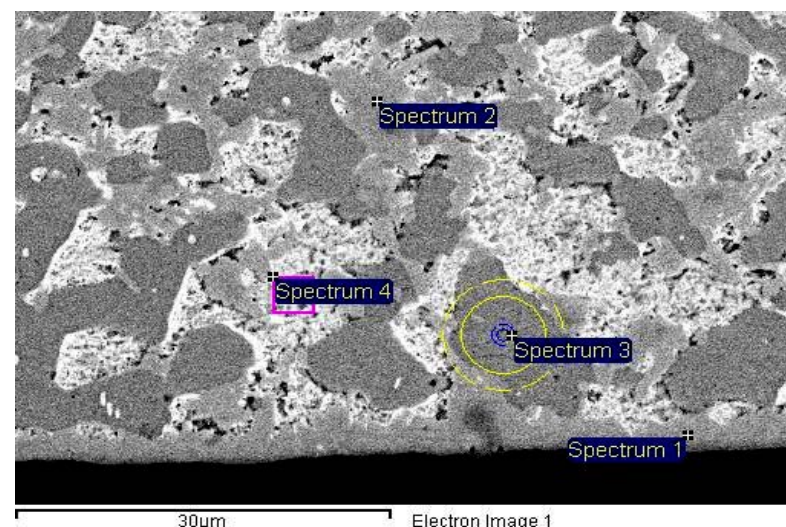
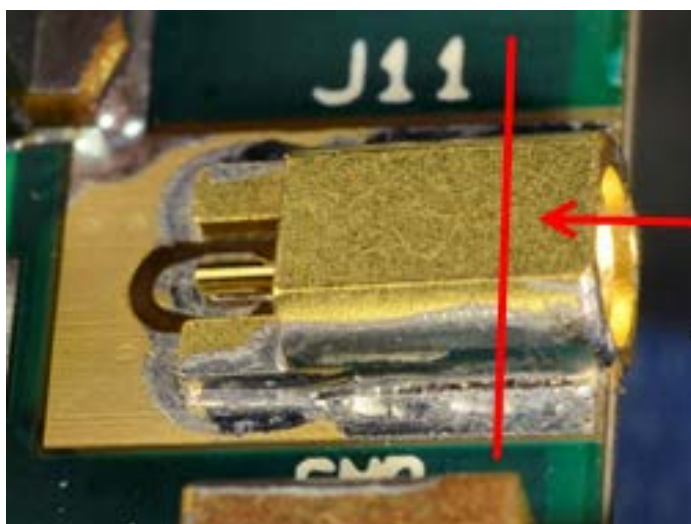


Figure 10. Soldered Surface Mount Connector with Joint Crack; Arrow Points to the Sectioning Plane. Close Up of Solder Section Attached to Circuit Board Pad (2200X).

Spectrum 2 is an Unknown Phase, Spectrum 3 is Tin, Spectrum 4 is Lead.



Manual Soldering with High Temperature

Case Study 3 Results

- Overall Joint Composition:
At the 3 to 4 Gold Weight
Percent Maximum Limit of
IPC-AJ-820A Assembly and
Joining Handbook; Nickel
Greater Than 1 Wt%

Table 1. Composition of the Overall Solder Joint in Figure 10

Element	Weight%	Atomic%
Ni K	1.24	2.85
Sn L	66.71	76.08
Au M	3.82	2.62
Pb M	28.23	18.45
Totals	100.00	100.00

- Per Spectrum 2's At% Data,
the Phase was Identified:
 $(\text{Au}_{0.45} \text{Ni}_{0.55})\text{Sn}_4$.

Table 2. Composition of the Phase Marked as Spectrum 2 in Figure 10

Element	Weight%	Atomic%
Ni K	5.39	10.93
Sn L	79.91	80.18
Au M	14.71	8.89
Totals	100.0	100.0

Its morphology is different from the typical acicular or platelet structure of the expected compound, AuSn_4 .

**1.2 Weight Percent Nickel in Joint and Nickel in Compound Revealed
Too High of a Manual Soldering Temperature**



Manual Soldering with High Temperature

Case Study 3 Results

- Four Corrective Actions
 - 1) Thinner Gold Finish on the Surface Mount Connector
 - 2) More Producing Flux
 - 3) Higher First Pass Yield, to Reduce Manual Rework
 - 4) More Heat Sink in Fixture Setup, to Lower Soldering Temperature
- The Present Analysis Shows High Temperature Can Cause Significant Nickel Dissolution in Addition to the Gold Dissolution. Unlike the Case of Embrittlement from AuSn_4 Gold-Tin Compound, There is No Industry Accepted Weight Percent Limit for Embrittlement with Gold-Nickel-Tin Compound.

Less Gold and Lower Temperature Improved Manual Soldering



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Solder Joint Embrittlement Understanding Aids Reliable Solder Joint Design



Hard Gold Surface Finish

Case Study 4 Data

- A Pin with a Hard Gold Over Nickel Finish was Soldered to a Circuit Board's Plated-Through-Hole with Sn63Pb37 alloy.

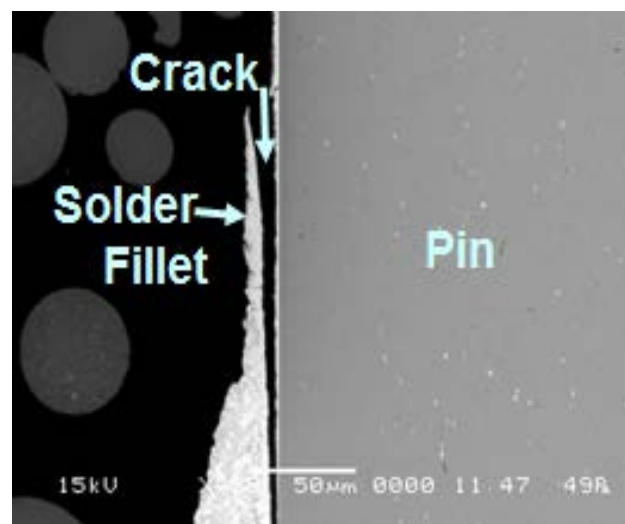
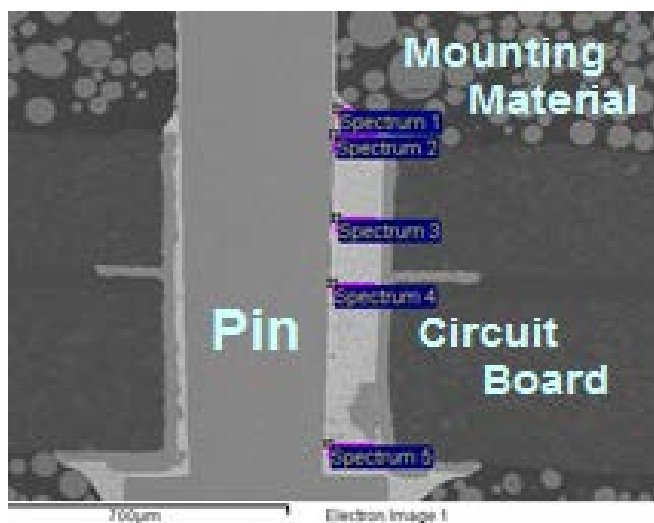


Figure 11. Cross-Sections of Solder Fillets after Pin Soldering (150X on Left). Close Up on Right Shows Fillet Separation from Pin and Residual Gold on Pin (275X).

Crack Cross-Section Shows Hard Gold Remaining on Pin after Soldering



Hard Gold Surface Finish

Case Study 4 Data

- Separation is Between Residual Gold Layer and AuSn_2 Layer which is Contiguous with AuSn_4 Layer.
- Additional AuSn_4 Compound is Distributed Throughout Fillet.

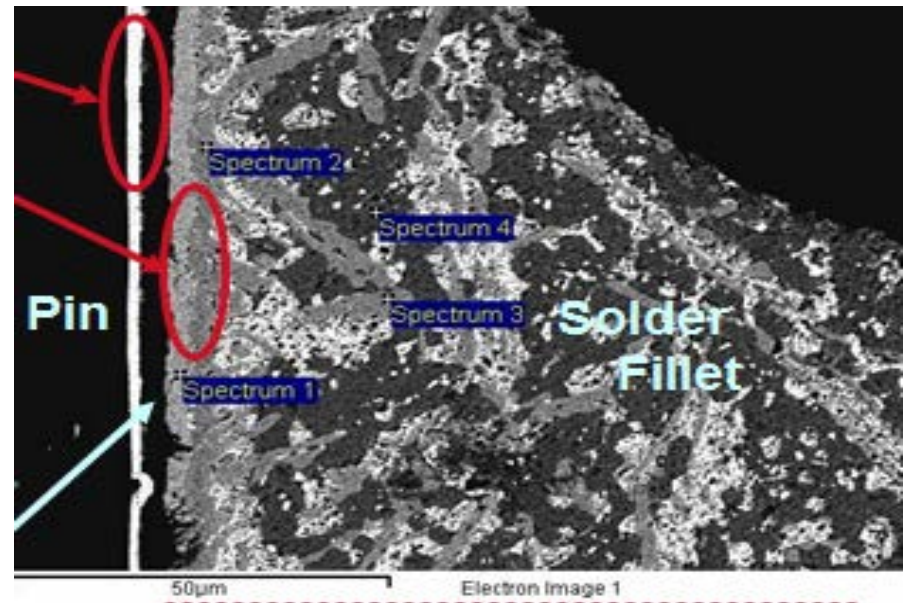


Figure 12. Microstructure of a Crack, Showing Residual Gold Layer (Top Oval), Crack (Lowest Arrow) and Duplex $\text{AuSn}_2/\text{AuSn}_4$ Compound Layer (Other Oval) in the Fillet (700X). The AuSn_2 is Closest to the Gold.

**Not All of the Gold was Dissolved from the Pin During Soldering.
The AuSn_2 Occurred Only As Part of the Duplex Interface Layer.**



Hard Gold Surface Finish

Case Study 4 Results

- Gold Dissolution and Reaction with Tin Formed AuSn_4 While the Solder was Molten.
- In Contrast, High Melting Point AuSn_2 Formed by Solid State Diffusion.
- Fast Diffusion (Warburton and Turnbull) Between Undissolved Gold and the AuSn_4 Occurred During Cooling of the Solidified Joint, and Formed the Adjacent AuSn_2 Layer.
- The Diffusion Also Weakened the Gold-to- AuSn_2 Interface with Vacancy Accumulation.
- Fillet Contraction Produced a Peeling Stress and Cracked the Weakened Interface.

Table 3. Gold Hardness and Purity Specifications

Gold Hardness	Gold Specification (Type describes Purity; Code describes Hardness)	Minimum Mass Percent Gold
Soft	ASTM B488-11, Type III, Code A	99.9
Hard	ASTM B488-11, Type II, Codes B, C & D	99.0
Soft & Hard	ASTM B488-11, Type I, Codes A, B & C	99.7

Plated Layers with Up to 20% Vacancies (Bhadeshia) Enable Solid State Diffusion and Accumulation of Vacancies in a Weakened/Voided Layer.



Hard Gold Surface Finish

Case Study 4 Results

- Hard Gold Has at Least 3 to 10 times the Impurity Level as Soft Gold (Table 3)
- Hard Gold Typically Contains Nickel, Iron or Cobalt Hardening Element
- At 230C, Nickel and Gold Dissolve at 0.05 and 100 Microinches per Second

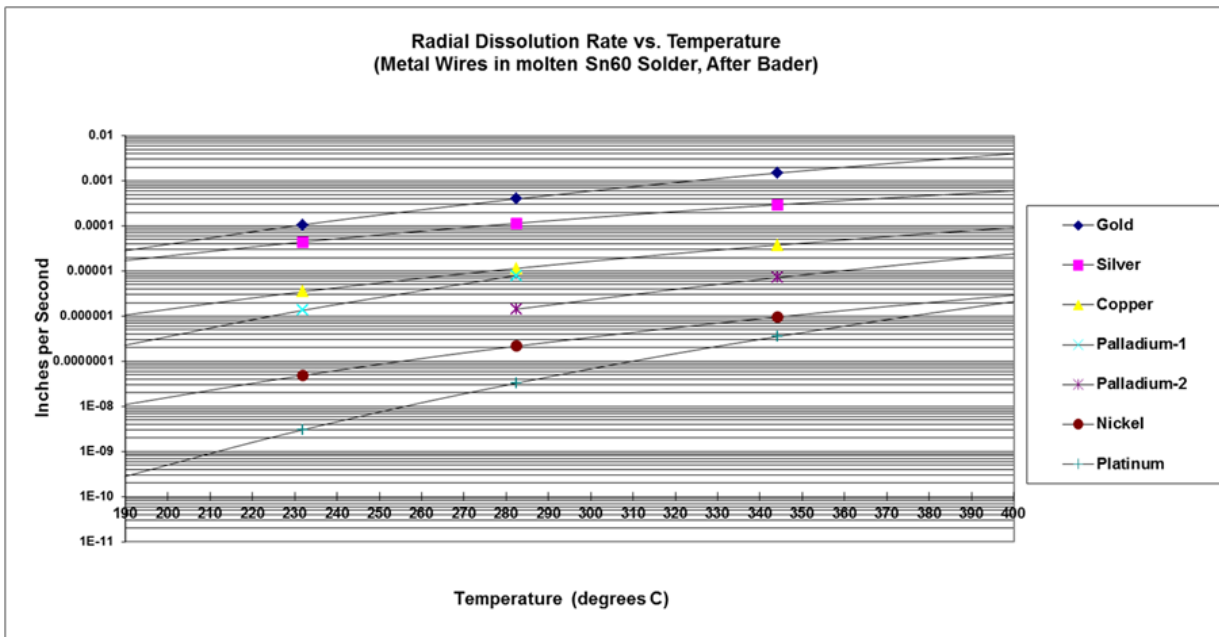


Figure 13. Radial Dissolution Rate vs. Temperature of Metal Wires in Molten Sn60Pb40 Alloy, After Bader (Chart Extrapolates Beyond Data Ranges).

Hardening of the Gold Slowed Its Dissolution , Enabled Its Diffusion & Caused AuSn₂ Formation and Cracking. Must Check for Hard Gold Removal.



**Radial Dissolution Rate vs. Temperature
(Metal Wires in molten Sn60 Solder, After Bader)**

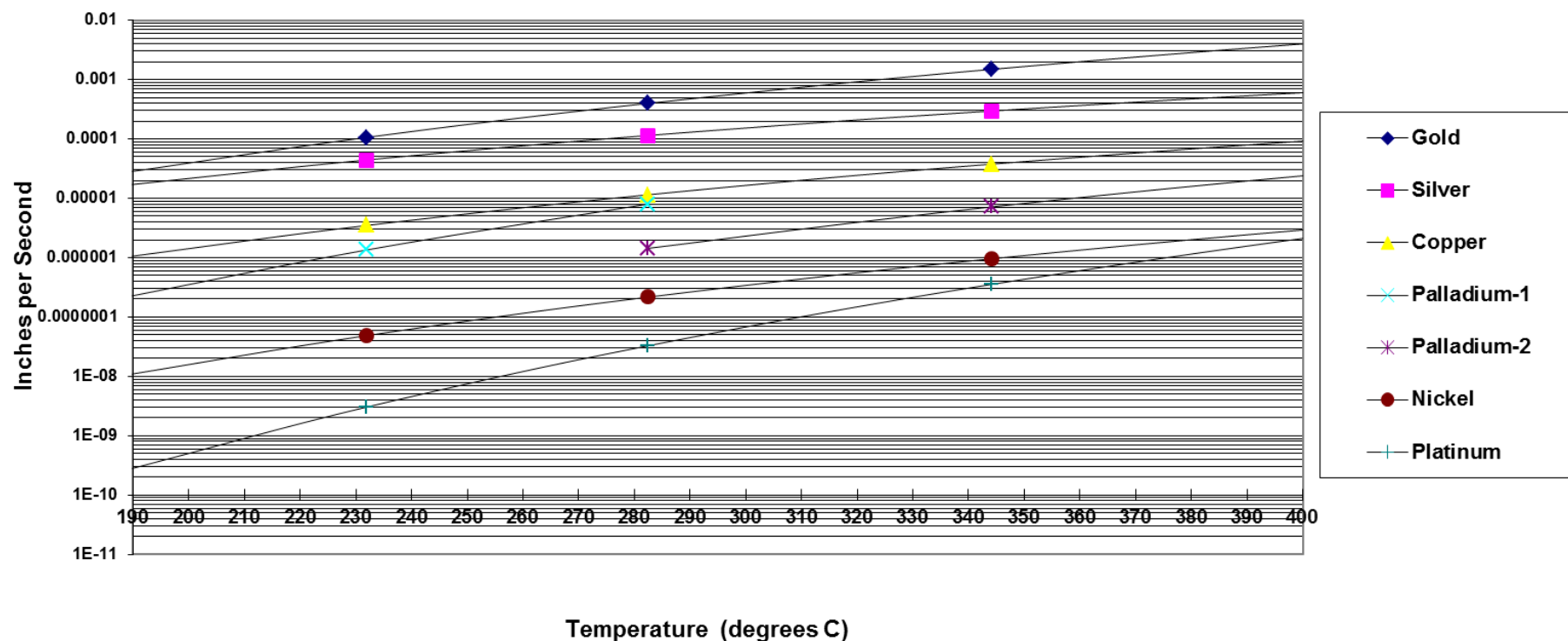


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 - Solder joint embrittlement is a decrease in solder joint durability, due to dissolution and/or reaction with a finish such as gold and/or palladium
- Four Case Study Examples of Embrittlement
 - Gold-Tin Compounds Precipitate onto an Interface
 - Excessive Gold in Soft Solder Joint
 - Manual Soldering with High Temperature
 - Hard Gold Surface Finish
- Standards: Weight %, Finish Thickness; Each Pb-Free Solder Alloy

Solder Joint Embrittlement Understanding Aids Reliable Solder Joint Design



Establishing Maximum Gold and Palladium Weight Percentages and a Calculator Equation

- IPC-AJ-820A Assembly and Joining Handbook Calls Out a 3 to 4 Weight Percent (Wt%) Maximum Gold Limit in a Soft Solder Joint
- 1 Wt% Au Increases Strength, 2.5 Wt% Au Improves Ductility (Wild, Sn63Pb37)
- Maximum Palladium (Pd) is 2 Wt% (Vianco)

	Wt%(Au)	=	$\frac{100\% \times [V(\text{Au}) \times D(\text{Au})]}{[V(\text{Au}) \times D(\text{Au})] + [V(\text{Sn63}) \times D(\text{Sn63})]}$
where			
	V(Au)	=	Volume of gold
	V(Sn63)	=	Volume of solder joint
	D(Au)	=	Density of gold
	D(Sn63)	=	Density of Sn63 solder
	Wt%(Au)	=	Weight percent gold in the solder joint

Figure 14. Equation for Calculating the Weight Percent of Gold in a Soft Solder Joint

Weight % Equation Easily is Modified for Lead-Free Solder Alloys and Palladium



Deriving Maximum Gold and Palladium Thicknesses for Solder Joint Embrittlement Prevention (SJEP)

- Individual Calculations Below for a Simple, Equal Area Solder Joint (e.g., 100 X 100 mils)
- The Maximum Gold and Palladium Were 4 and 2 Wt% in Sn63Pb37
- Four Bolded Rectangles Show Where the Maximum was Nearly Reached
- Gold 0.45 μm [18 microinches] or less on each side of an “equal area” solder joint, i.e., on both a surface mount component and a circuit board, or palladium 0.35 μm [14 microinches] or less on each side of an “equal area” solder joint, are acceptable when the gold is soft and pure per ASTM B488-11, Type III, Code A, and when the solder joint is at least 2.0 mils thick (equivalent to a 4 mil thick stencil).

1. Thin Gold and Palladium Calculations

1.1 Weight Percent Gold

Solder Joint Thickness (mils)	Microinches Gold on Component with zero			Microinches Gold on Substrate			Microinches Gold on Component with same			Microinches Gold on Substrate		
	8	15	18	0	0	0	8	15	18	8	15	18
1.0	1.78	3.29	3.93				3.51	6.38	7.56			
2.0	0.90	1.67	2.00				1.78	3.29	3.93			
3.0	0.60	1.12	1.34				1.20	2.22	2.65			
4.0	0.45	0.84	1.01				0.90	1.67	2.00			

1.2 Weight Percent Palladium set at 50% of the Weight Percent Gold

Solder Joint Thickness (mils)	Microinch Palladium on Component with 0			Microinches Pd on Substrate			Microinches Pd on Component with same			Microinches Palladium on Substrate		
	6.4	11.9	14.2	0	0	0	6.3	11.7	13.9	6.3	11.7	13.9
1.0	0.89	1.65	1.97				1.75	3.19	3.78			
2.0	0.45	0.84	0.99				0.88	1.62	1.93			
3.0	0.30	0.56	0.67				0.59	1.09	1.29			
4.0	0.22	0.42	0.50				0.44	0.82	0.97			

Figure 15. Calculated Wt % Gold and Palladium for Selected Solder and Finish Designs

Joint Designs and Weight % Limits Determine Maximum Plating Thicknesses



Considering the Possibility of Gold Thickness Range and Lead-Free Solder Alloy Weight % Limits

- Figure 1 Showed Porosity vs. Gold Thickness. It Implied a Minimum Electrolytic Gold Thickness to Ensure Solderability

Table 4. Example of How to Specify Electrolytic Gold Thickness Range for Solderability and Solder Joint Embrittlement Prevention (SJEP)

Maximum Gold Weight Percent in Solder Joint	Solidified Solder Thickness (mils)	Minimum Gold Thickness for Solderability (microinches)	Calculated Maximum Gold Thickness for SJEP (microinches)
4.0	3.0	20	54
3.0	3.0	20	40
4.0	2.0	20	36
3.0	2.0	20	27

- Based on Their Value to Prevent Solder Joint Embrittlement, We Need to Determine Maximum Weight Percent Limits for Each Lead-Free Solder Alloy Used in the Electronics Assembly Industry, Using Properties Such As Impact Strength (Bester) and Fracture Elongation Vs. Gold Weight Percent.

Gold Thickness Ranges and Lead-Free Solder Alloy Weight % Limits are Needed



Summary (1 of 2)

Why We Did the Work, a Definition, Embrittlement Fundamentals

Lead-Free Finishes Are Risky, Not Just Tin (whiskers) but also Gold (embrittlement).
Solder Joint Embrittlement is Defined as a Decrease in Solder Joint Durability Due to Dissolution and/or Reaction with a Finish Such as Gold and/or Palladium.
An Understanding of Solder Joint Embrittlement Aids Reliable Solder Joint Design.
Soldering Finish, Pb-Free Alloy, Process and Compounds can Cause Embrittlement.
Maximum Gold Thickness Needs to be Known to Prevent Embrittlement.

Gold-Tin Compound Precipitates onto Interface - Case Study 1

Avoid “Infinite” Source of Gold Available to Dissolve Into Solder Joint.
Embrittlement Mechanism: Gold from Substrate Precipitated Onto Pin.

Excessive Gold in Soft Solder Joint - Case Study 2

10 Weight Percent Gold is Too High for SnAg3.7 Soldered Connector Pin.
Optimal Levels for Each Experimental Factor Reduced the Pin and Board Gold,
Increased the Solder Volume and Interacted to Increase Pin Pull Strength.
Solder Joint Embrittlement can be Prevented by Process and Design.



Summary (2 of 2)

Manual Soldering with High Temperature - Case Study 3

Surface Mount Connector with Joint Crack Had $(\text{Au}_{0.45} \text{Ni}_{0.55})\text{Sn}_4$ Compound.
Overall Joint Composition had 3.8 Wt% Gold and 1.2 Wt% Nickel, Revealing Too High a Manual Soldering Temperature.
Less Gold and Lower Temperature Improved Manual Soldering.

Hard Gold Surface Finish - Case Study 4

Cross-Section of Joint Crack Revealed Hard Gold Remaining on Pin After Soldering.
The AuSn_2 Next to the Crack Occurred Only As Part of a Duplex Layer with AuSn_4 .
Plated Layers with Up to 20% Vacancies (Bhadeshia) Enable Solid State Diffusion and Accumulation of Vacancies in a Weakened/Voided Layer.
Hardening of the Gold Slowed Its Dissolution Rate, Enabled It to Diffuse with AuSn_4 and Caused the AuSn_2 Formation & Cracking. Must Check for Hard Gold Removal.

Standards Need Weight % & Finish Thickness for Each Solder Alloy

Weight % Equation Easily is Modified for Lead-Free Solder Alloys and Palladium.
Joint Designs and Weight % Limits Determine Maximum Plating Thicknesses
Gold Thickness Ranges and Lead-Free Solder Alloy Weight % Limits are Needed



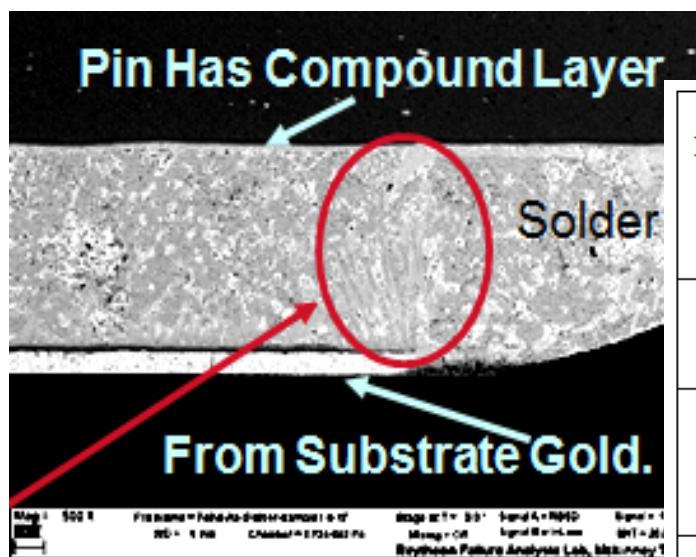
Acknowledgements

The author gratefully acknowledges the company for permission to publish the paper, as well as the following sponsors, collaborators and analysts:

Keith Kirchner, Bill Ortloff, Karl Worthen, John Bedinger, George Barson,
David Pinsky, Anthony Rafanelli, Chris Bender, Jason Bain, Karla Willis,
Emily Hoffman, Robert Champaign, Kennedy Coleman, Terry Banks, Shirley Smith,
Dung Truong, Terry Graham, Marti Fountain, Theresa Hendricks, Michael Dwight and
Marlin Downey.

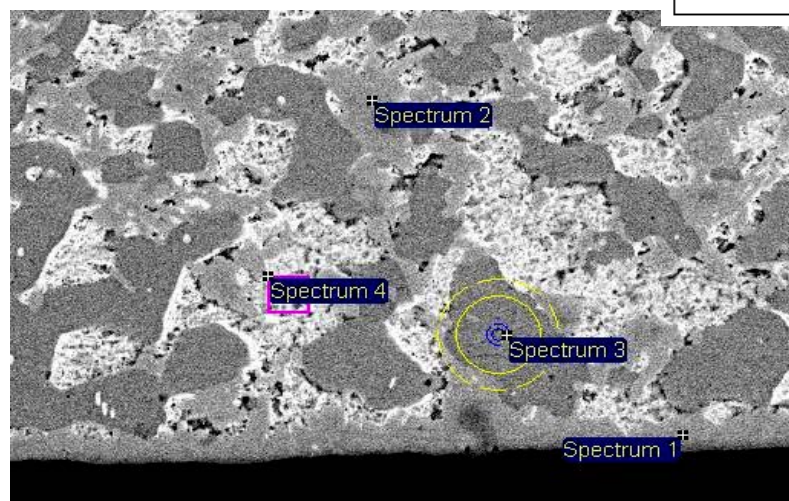
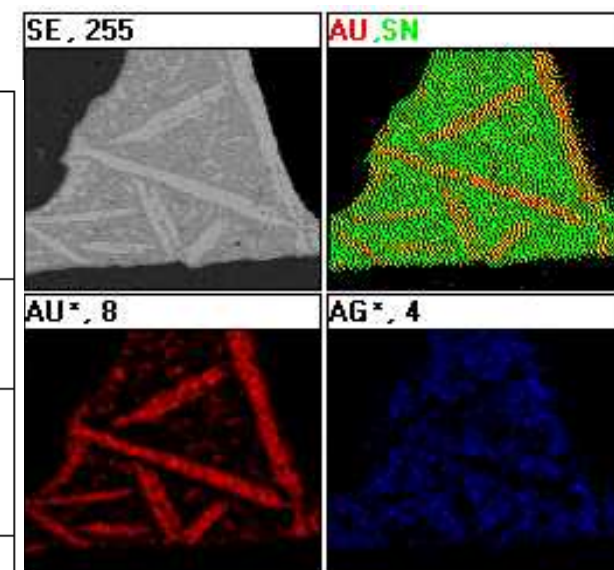


Maximum Gold Weight Percent in Solder Joint	Solidified Solder Thickness (mils)	Minimum Gold Thickness for Solderability (microinches)	Calculated Maximum Gold Thickness for SJEP
4.0	3.0	20	54
3.0	3.0	20	40
4.0	2.0	20	36
3.0	2.0	20	27



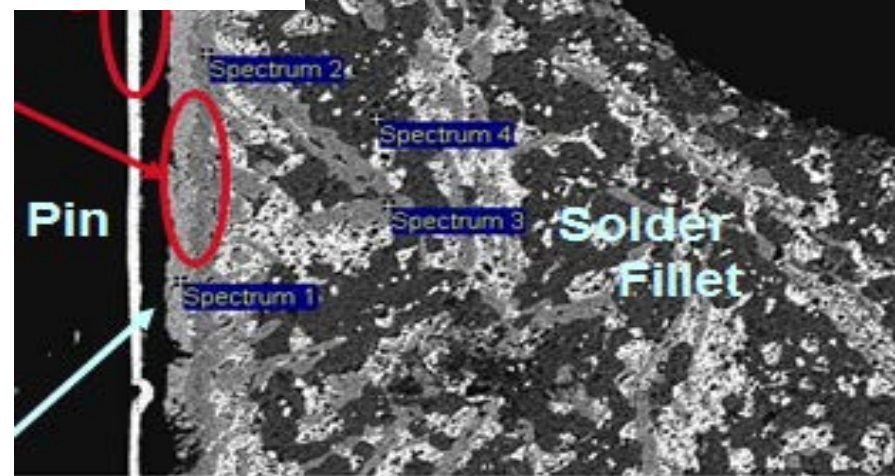
Questions?

Gold Hardness	Gold Specification (Type describes Purity; Code describes Hardness)	Minimum Mass Percent Gold
Soft	ASTM B488-11, Type III, Code A	99.9
Hard	ASTM B488-11, Type II, Codes B, C & D	99.0
Soft & Hard	ASTM B488-11, Type I, Codes A, B & C	99.7



30µm

Electron Image 1



50µm

Electron Image 1