PCB SURFACE FINISHES & THE CLEANING PROCESS - A COMPATIBILITY STUDY

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ABSTRACT

All PCBs that are manufactured require a surface finish to protect exposed copper on the surface which if left unprotected, can oxidize, rendering the board unusable. To address this issue, it is common to surface treat the PCB prior to assembly and reflow. The surface finish not only prevents oxidation of the underlying copper, but guarantees a solderable surface. A cost effective and widely used approach to PCB surface finish is HASL (Hot Air Solder Leveling). However, as circuit complexity and component density have increased, HASL has reached its limitations, necessitating the need for thinner coatings. Thus, coatings such as Immersion Tin (ImSn), Immersion Silver (ImAg), Organic Solderability Preservatives (OSP), and Electroless Nickel Immersion Gold (ENIG) are becoming more widely used.

As most PCBs designed for use in high reliability applications are cleaned or 'washed' in aqueous-based cleaning systems, the effect of the cleaning solution on the surface finish is of great concern. Depending on the cleaning process employed, it is possible for stains to appear on the plating or in the worst case, for the plating to be completely stripped from the PCB rendering the applied surface finish useless.

This study was designed to investigate the effect of reflow and various cleaning agent types on ImSn, ImAg and ENIG surface finishes. Unpopulated ZESTRON[®] test vehicles, with the appropriate surface finish, were used for all trials. In a previous study by the authors, the OSP surface finish was analyzed for integrity within an aqueous-based cleaning system and was therefore excluded from this study.

An inline spray-in-air cleaning process was chosen to assess the surface finish integrity. Three (3) aqueous cleaning agent types were selected for use within this study. Two (2) alkaline cleaning agents, inhibited and uninhibited, as well as a pH neutral cleaning agent, were used. Cleaning system process variables were established and held constant for all trials. Surface finish assessment following reflow and cleaning was conducted using visual inspection, adhesion test, copper test (ImAg and ImSn), nickel test (ENIG), and the X-Ray Fluorescence (XRF) test. Additionally, baseline tests were conducted on boards with all finish types without exposure to reflow or the cleaning process in order to assess the effect of the reflow process.

KEYWORDS

PCB surface finish, cleaning process, Surface test analysis, copper test, nickel test, adhesion test, XRF test

INTRODUCTION

Given the complexity of PCB designs today, including the use of stacked components, shrinking standoffs, and increased board density to name a few, cleaning for high reliability applications remains a critical requirement for OEMs and ECMs alike. Typically, engineered aqueousbased cleaning agents are used primarily in spray-in-air cleaning processes. In order to protect the copper surfaces from oxidizing, various surface finishes are applied to the bare board. In recent years, PCB surface treatments have moved away from HASL finishes and more and more are treated with ImSn, ImAg, OSP, and ENIG. Thus, understanding the compatibility of cleaning agent types with the various surface finish treatments is critical.

As surface finish treatments have evolved, so too have the technologies of engineered cleaning agents. Early formulations were surfactant based, relatively high pH, alkaline cleaning agents without the benefit of inhibition packages to prevent material compatibility issues with the substrate and components. Cleaning agent development continued with the formation of dynamic surfactant and micro phase based products. Many of these are still alkaline, however, some include inhibition packages. More recently, pH neutral formulations with inhibition packages were developed.

Thus, electronics manufacturers that are using an aqueousbased cleaning process may be using inhibited or uninhibited alkaline cleaning agents and/or pH neutral cleaning agents. It is possible that certain cleaning agent types can stain the plating and/or possibly strip the coating completely. Thus, assessing the compatibility of various surface finishes with the various cleaning agent types is of interest within the industry.

Previously, the authors investigated the effect of reflow and cleaning on OSP surface finish and so this was excluded from this study [1].

The objective of this study was to investigate the effect of both the reflow and cleaning process on designated surface finishes. For the cleaning process, inhibited and uninhibited dynamic surfactant and micro phase cleaning agents were selected.

The analytical test methods used to assess the effect on the surface finish included visual inspection, adhesion and X-ray Fluorescence (XRF) tests for all surface finish types, copper test for ImAg and ImSn only and nickel test for ENIG only.

METHODOLOGY

Upon developing the Design of Experiment (DOE), three (3) key variables were identified for possible effect on the selected surface finishes. These were reflow, cleaning agent type and cleaning agent exposure time to the specific surface finish. For the selected variables, all surface finish types were subjected to two (2) reflow conditions, four (4) cleaning agent conditions and two (2) wash process conditions. Reference Table 1.

Table 1. Variables on Surface Finishes

	Process Variable	Surface Finish		
1	Reflow			
1	No Reflow			
	Cleaning Agent A			
2	Cleaning Agent B	ImAg	ENIC	ImSn
2	Cleaning Agent C	miAg	LINIO	mon
	Cleaning Agent D			
3	3 Wash Passes			
5	6 Wash Passes			

For this study, the authors chose the ZESTRON[®] test vehicle as the substrate. However, no solder paste or components were used. The required number of substrates were sourced from the supplier complete with the designated finish. Reference Figure 1.



Figure 1. ZESTRON® Test Vehicle

The DOE was developed to challenge the evaluation. Thus, with regard to reflow, a lead-free profile was selected due to its higher peak temperature, thereby increasing potential impact on the surface finishes. However, to fully understand the impact of the reflow process as it relates to the cleaning process, boards that were and were not reflowed were each subjected to the cleaning process. Reference Table 2 in the Appendix for the reflow oven zone temperatures and the profile curve.

With regard to the cleaning process, the authors utilized a spray-in-air inline cleaning process and chose to subject the boards to multiple passes. It is common in many PCB assembly applications that substrates are processed through multiple heat and cleaning cycles. Again, to challenge the evaluation, the authors subjected the boards to three (3) and six (6) consecutive cleaning cycles respectively.

Four (4) cleaning agents were used for each cleaning cycle to assess their impact on each surface finish. Cleaning agent types used were:

- Cleaning Agent A: Uninhibited alkaline dynamic surfactant
- Cleaning Agent B: Uninhibited alkaline micro phase
- Cleaning Agent C: Inhibited alkaline dynamic surfactant
- Cleaning Agent D: Inhibited pH neutral micro phase

The inline cleaning system process settings selected represent the industry average based on the authors' experience. These settings were held constant for all trials. Reference Table 3.

Table 3. Cleaning Process Parameters

Wash Stage			
Equipment	Inline Cleaner		
Cleaning Agent Concentration	20%		

Conveyor Belt Speed	1 ft/min		
Pre-Wash Pressure	50 DSI / 40 DSI		
(Top/Bottom)	50 PSI / 40 PSI		
Wash Spray	8-spray bar standard		
Configuration	intermix		
Wash Pressure	00 BSI / 70 BSI		
(Top/Bottom)	90 PS1 / 70 PS1		
Wash Hurricane Pressure	40 BSI / 40 BSI		
(Top/Bottom)	40 F31 / 40 F31		
Wash Temperature	150°F / 65.5°C		
Rinsin	g Stage		
Rinsing Agent	DI-water		
Rinse Pressure			
Rinse Pressure (Top/Bottom)	90 PSI / 80 PSI		
Rinse Pressure (Top/Bottom) Rinse Hurricane Pressure	90 PSI / 80 PSI		
Rinse Pressure (Top/Bottom) Rinse Hurricane Pressure (Top/Bottom)	90 PSI / 80 PSI 75 PSI / 45 PSI		
Rinse Pressure (Top/Bottom)Rinse Hurricane Pressure (Top/Bottom)Rinse Temperature	90 PSI / 80 PSI 75 PSI / 45 PSI 140°F / 60°C		
Rinse Pressure (Top/Bottom)Rinse Hurricane Pressure (Top/Bottom)Rinse TemperatureFinal Rinse Pressure	90 PSI / 80 PSI 75 PSI / 45 PSI 140°F / 60°C 25 PSI / 25 PSI		
Rinse Pressure (Top/Bottom)Rinse Hurricane Pressure (Top/Bottom)Rinse TemperatureFinal Rinse PressureFinal Rinse Temperature	90 PSI / 80 PSI 75 PSI / 45 PSI 140°F / 60°C 25 PSI / 25 PSI Room Temperature		
Rinse Pressure (Top/Bottom)Rinse Hurricane Pressure (Top/Bottom)Rinse TemperatureFinal Rinse PressureFinal Rinse TemperatureDrying	90 PSI / 80 PSI 75 PSI / 45 PSI 140°F / 60°C 25 PSI / 25 PSI Room Temperature 5 Stage		
Rinse Pressure (Top/Bottom)Rinse Hurricane Pressure (Top/Bottom)Rinse TemperatureFinal Rinse PressureFinal Rinse TemperatureDryingDrying Method	90 PSI / 80 PSI 75 PSI / 45 PSI 140°F / 60°C 25 PSI / 25 PSI Room Temperature g Stage Hot Circulated Air		

Given the variables that would be assessed, a test matrix was developed requiring 48 trials. Reference Table 4.

Table 4.	Cleaning	Trial	Conditions

	Surface		Cleaning	# Wash
Trial #	Finish	Reflow	Agent	Passes
1	ImAg	No	A	3
2	ImAg	No	Δ	6
3	ImAg	Yes	<u>А</u>	3
1	ImAg	Ves	Δ	6
5	ENIG	No		3
6	ENIC	No		5
7	ENIC	Vas	A	2
0	ENIC	I es	A	5
0	ENIG	I es	A	0
9	ImSn	NO	A	3
10	ImSn	No	A	6
11	ImSn	Yes	A	3
12	ImSn	Yes	A	6
13	ImAg	No	В	3
14	ImAg	No	В	6
15	ImAg	Yes	В	3
16	ImAg	Yes	В	6
17	ENIG	No	В	3
18	ENIG	No	В	6
19	ENIG	Yes	В	3
20	ENIG	Yes	В	6
21	ImSn	No	В	3
22	ImSn	No	В	6
23	ImSn	Yes	В	3
24	ImSn	Yes	В	6
25	ImAg	No	С	3
26	ImAg	No	С	6
27	ImAg	Yes	С	3
28	ImAg	Yes	С	6

29	ENIG	No	С	3
30	ENIG	No	C	6
31	ENIG	Yes	C	3
32	ENIG	Yes	C	6
33	ImSn	No	C	3
34	ImSn	No	C	6
35	ImSn	Yes	C	3
36	ImSn	Yes	C	6
37	ImAg	No	D	3
38	ImAg	No	D	6
39	ImAg	Yes	D	3
40	ImAg	Yes	D	6
41	ENIG	No	D	3
42	ENIG	No	D	6
43	ENIG	Yes	D	3
44	ENIG	Yes	D	6
45	ImSn	No	D	3
46	ImSn	No	D	6
47	ImSn	Yes	D	3
48	ImSn	Yes	D	6

Each trial included three (3) boards. Thus, 48 boards were sourced for each finish type yielding a total of 144 boards sourced for the complete DOE evaluation. Of the three (3) boards prepared for each trial, the surface finish was analyzed as follows:

- Board 1: Adhesion test and visual inspection: all surface finishes
- Board 2: Surface analysis nickel test: ENIG only; copper test: ImAg and ImSn only
- Board 3: XRF test: all surface finishes

Also, three (3) additional boards of each surface finish were sourced for baseline analysis for the copper, nickel and XRF tests. Baseline measurements were made on control boards under two conditions:

- Not cleaned, no reflow
- Not cleaned, reflow

In this way, the authors were able to evaluate the impact on each surface finish resulting from reflow, cleaning agent type and wash process exposure time as compared to baseline values.

As a point of information, Board 1 and Board 2 analyses were conducted at the ZESTRON Technical Center whereas the Board 3 analysis was conducted by an independent certified lab.

SURFACE ANALYSIS PROTOCOL Visual Inspection

Visual inspection was conducted on Board 1 for all trials using a Keyence VHX-1000 microscope. Each board was inspected under 50-100x magnification and any surface defect such as scratches or stains were noted.

Adhesion Test

The adhesion test was conducted on Board 1 for all trials and in accordance with IPC TM-650 Method 2.4.1. The tape used was 3M Type 600, 0.5" wide and applied at three locations on the substrate. Reference Figure 2.



Figure 2. Areas used for adhesion test

The tape was applied to the areas indicated in Figure 2. The tape was then peeled off and the board surface and inspected for peeling of any plating material. Reference Figure 3.



Figure 3. Adhesion test tape peel

Surface Test

The surface tests were conducted utilizing test kits from EMD Millpore Corporation. These tests utilize test strips that react with the metallic surface to indicate the presence of nickel and copper. A pink color indicates the presence of nickel and a violet color indicates the presence of copper. Reference Figure 4-5 for the representative scale in mg/l Cu (ppm equivalent) and mg/l Ni (ppm equivalent).

Copper Test (ImSn and ImAg boards only)

The copper test was conducted on Board 2 for all ImSn and ImAg trials. This test measures both copper (II) and copper (I) ions. The copper concentration is measured semiquantitatively by visual comparison of the reaction zone on the test strip with the fields of a color scale [2]. Reference Figure 4. The copper test strips were sourced from EMD Millipore and conducted in accordance with the supplier recommended procedure. Reference Figure 6 for the locations on the board that were tested. One test strip was used for each sample area, labeled A, B, C, and D.



Figure 4. Representative scale - Copper Test

Following the supplier recommended procedure, a test strip was wetted with DI-water and then pressed lightly on the surface to be tested for 10 seconds. The strip was then inspected after 30 seconds. A color change of the test strip from white to pink indicates presence of copper. The intensity of the color as compared to the reference scale indicates the approximate concentration (ppm) of copper in that area of the board. A new strip was used for each area tested.

Nickel Test (ENIG boards only)

The nickel test was conducted on Board 2 for all ENIG trials. The nickel concentration is measured semiquantitatively by visual comparison of the reaction zone on the test strip with the fields of the color scale [3]. Reference Figure 5. Nickel test strips were sourced from EMD Millipore and conducted in accordance with the supplier recommended procedure. Reference Figure 6 for the locations on the board that were tested. One test strip was used for each sample area, labeled A, B, C, and D.



Figure 5. Representative Scale – Nickel Test

This test required a 25% ammonia solution which was also sourced from EMD Millipore. Following the supplier recommended procedure, a test strip was wetted with the ammonia solution and then pressed lightly on the surface to be tested for 10 seconds. It was inspected after 30 seconds. A color change of the test strip from white to pink as compared to the reference scale indicates presence of nickel. The intensity of the color indicates the approximate concentration (ppm) of nickel in that area of the board. A new strip was used for each area tested.



Figure 6. Areas used for copper and nickel surface tests

XRF Test (all boards)

The XRF test was conducted on Board 3 for all trials. This test was used to measure the plating thickness of all finishes on all boards and conducted by an independent certified lab. Thickness measurements were made for each surface finish:

- ImAg Thickness of silver plating
- ImSn Thickness of tin plating
- ENIG Thickness of gold and nickel plating

The measurements were made on six (6) areas of each board and then averaged. These values were then compared with those of the baseline tests. The areas used for the measurements are detailed in Figure 7.



Figure 7. Areas used for XRF Tests

RESULTS

Results – Visual Inspection

Results of the visual inspection on the Immersion Silver plated boards are detailed in Table 5.

Table 5. Visual Inspection Results - ImAg Plating

				0	0
Trial #	Surface Finish	Reflow	Cleaning Agent	# Wash Passes	Visual Inspection Results
1	ImAg	No	А	3	0
2	ImAg	No	А	6	-
3	ImAg	Yes	А	3	+
4	ImAg	Yes	А	6	0
13	ImAg	No	В	3	++
14	ImAg	No	В	6	+
15	ImAg	Yes	В	3	++
16	ImAg	Yes	В	6	++
25	ImAg	No	С	3	++
26	ImAg	No	С	6	++
27	ImAg	Yes	С	3	++
28	ImAg	Yes	С	6	++
37	ImAg	No	D	3	++
38	ImAg	No	D	6	++
39	ImAg	Yes	D	3	++
40	ImAg	Yes	D	6	++

++: No effect

+: Slight effect

0: Plating removed in some areas

-: Plating removed in most areas

Upon reviewing these results, the authors inferred the following:

- Cleaning Agent A (dynamic surfactant, uninhibited) adversely affected the Immersion Silver plating. The plating removal was most severe for trials 2 (no reflow - 6 passes) and 4 (reflow - 6 passes). Reference Figures 7A and 7B.
- Cleaning Agent C (dynamic surfactant, inhibited) had no affect under all conditions. Reference Figures 8A and 8B.
- Cleaning Agent D (pH neutral, inhibited) had no affect under all conditions. Reference Figures 8C and 8D.
- Cleaning Agent B (micro phase, uninhibited) had a slight affect for Trial 14 (no reflow 6 passes). Reference Figure 9.

Furthermore, all boards were exposed to air for an additional three weeks to check for any changes to the plating materials. No changes were noted and the plating materials showed no further degradation.



Figure 7A. Visual Inspection, Cleaning Agent A – Trial 2: No Reflow, 6 Passes



Figure 7B. Visual Inspection, Cleaning Agent A – Trial 4: Reflow, 6 Passes



Figure 8A. Visual Inspection, Cleaning Agent C – Trial 26: No Reflow, 6 Passes



Figure 8B. Visual Inspection, Cleaning Agent C – Trial 28: Reflow, 6 Passes



Figure 8C. Visual Inspection, Cleaning Agent D – Trial 38: No Reflow, 6 Passes



Figure 8D. Visual Inspection, Cleaning Agent D – Trial 40: Reflow, 6 Passes



Figure 9. Visual Inspection, Cleaning Agent B – Trial 14: No Reflow, 6 Passes

Results – Visual Inspection

Results of the visual inspection test on the Immersion Tin plated boards are summarized in Table 6.

Table 6. Visua	l Inspection	Results -	ImSn	Plating
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Trial #	Surface Finish	Reflow	Cleaning Agent	# Wash Passes	Visual Inspection Results
9	ImSn	No	А	3	++
10	ImSn	No	А	6	++
11	ImSn	Yes	А	3	++
12	ImSn	Yes	А	6	++
21	ImSn	No	В	3	++
22	ImSn	No	В	6	++
23	ImSn	Yes	В	3	++
24	ImSn	Yes	В	6	++

33	ImSn	No	С	3	++
34	ImSn	No	С	6	++
35	ImSn	Yes	С	3	++
36	ImSn	Yes	С	6	++
45	ImSn	No	D	3	++
46	ImSn	No	D	6	++
47	ImSn	Yes	D	3	++
48	ImSn	Yes	D	6	++

++: No effect

+: Slight effect

0: Plating removed in some areas

-: Plating removed in most areas

Upon reviewing these results, the authors inferred that all the cleaning agents were fully compatible with the Immersion Tin plating regardless of whether the boards were subjected to reflow.

Furthermore, the boards were exposed to air for an additional three weeks to check for any changes to the plating materials. No changes were noted and the plating materials showed no further degradation.

As the results were consistent for all variables evaluated, representative pictures from the most challenging process variables, 6 (six) passes, reflow and Cleaning Agent A (dynamic surfactant, uninhibited) are detailed in Figures 10A and 10B.



Figure 10A. Visual Inspection, Cleaning Agent A – Trial 10: No Reflow, 6 Passes



Figure 10B. Visual Inspection, Cleaning Agent A – Trial 12: Reflow, 6 Passes

Results – Visual Inspection

Results of the visual inspection on the Electroless Nickel Immersion Gold plated boards are summarized in Table 7.

Table 7. Visual Inspection Results - ENIG Plating

Trial #	Surface Finish	Reflow	Cleaning Agent	# Wash Passes	Visual Inspection Results
5	ENIG	No	А	3	++
6	ENIG	No	А	6	++
7	ENIG	Yes	А	3	++
8	ENIG	Yes	А	6	++
17	ENIG	No	В	3	++
18	ENIG	No	В	6	++
19	ENIG	Yes	В	3	++
20	ENIG	Yes	В	6	++
29	ENIG	No	С	3	++
30	ENIG	No	С	6	++
31	ENIG	Yes	С	3	++
32	ENIG	Yes	С	6	++
41	ENIG	No	D	3	++
42	ENIG	No	D	6	++
43	ENIG	Yes	D	3	++
44	ENIG	Yes	D	6	++

++: No effect

+: Slight effect

0: Plating removed in some areas

-: Plating removed in most areas

Upon reviewing these results, the authors inferred that all the cleaning agents were fully compatible with the ENIG plating regardless of whether the boards were subjected to reflow.

Furthermore, the boards were exposed to air for an additional three weeks to check for any changes to the plating materials. No changes were noted and the plating materials showed no further degradation.

As the results were consistent for all variables evaluated, representative pictures from the most challenging process variables, six (6) passes, reflow and Cleaning Agent A (dynamic surfactant, uninhibited) are detailed in Figure 11A and 11B.



Figure 11A. Visual Inspection, Cleaning Agent A – Trial 6: No Reflow, 6 Passes



Figure 11B. Visual Inspection, Cleaning Agent A – Trial 8: Reflow, 6 Passes

Results – Adhesion Test

Results of the adhesion test performed as per IPC TM-650 Method 2.4.1 on the Immersion Silver plated boards are summarized in Table 8.

Table 8.	Adhesion	Test	Results -	- ImAg	Plating
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Trial #	Surface Finish	Reflow	Cleaning Agent	# Wash Passes	Adhesion Test Results
1	ImAg	No	А	3	++
2	ImAg	No	А	6	++
3	ImAg	Yes	А	3	++
4	ImAg	Yes	А	6	++
13	ImAg	No	В	3	++
14	ImAg	No	В	6	++
15	ImAg	Yes	В	3	++
16	ImAg	Yes	В	6	++
25	ImAg	No	С	3	++
26	ImAg	No	С	6	++
27	ImAg	Yes	С	3	++
28	ImAg	Yes	С	6	++
37	ImAg	No	D	3	++
38	ImAg	No	D	6	++
39	ImAg	Yes	D	3	++
40	ImAg	Yes	D	6	++

++: No effect

+: Slight effect

0: Plating removed in some areas

-: Plating removed in most areas

Regardless if the substrates were subjected to reflow, all cleaning agents had no impact on any of the surface finishes. However, it is interesting to note that Cleaning Agent A (dynamic surfactant, uninhibited) had no impact on the surface finish as noted with the adhesion test even though it did impact the surface finish as evidenced through visual inspection. Reference Table 5.

Results – Adhesion Test

Results of the adhesion test performed as per IPC TM-650 Method 2.4.1 on the Immersion Tin plated boards are summarized in Table 9.

Table 9. Adhesion Test Results - ImSn Plating

Trial #	Surface Finish	Reflow	Cleaning Agent	# Wash Passes	Adhesion Test Results
9	ImSn	No	А	3	++
10	ImSn	No	А	6	++
11	ImSn	Yes	А	3	++
12	ImSn	Yes	А	6	++
21	ImSn	No	В	3	++
22	ImSn	No	В	6	++
23	ImSn	Yes	В	3	++
24	ImSn	Yes	В	6	++
33	ImSn	No	С	3	++
34	ImSn	No	С	6	++
35	ImSn	Yes	С	3	++
36	ImSn	Yes	С	6	++
45	ImSn	No	D	3	++
46	ImSn	No	D	6	++
47	ImSn	Yes	D	3	++
48	ImSn	Yes	D	6	++

++: No effect

+: Slight effect

0: Plating removed in some areas

-: Plating removed in most areas

Regardless of reflow, and number of wash passes, none of the cleaning agents impacted the surface finish.

Results – Adhesion Test

Results of the adhesion test performed as per IPC TM-650 Method 2.4.1 on the Electroless Nickel Immersion Gold plated boards are summarized in Table 10.

Trial	Surface	Doflow	Cleaning	# Wash	Adhesion
#	Finish	Kenow	Agent	Passes	Test Results
5	ENIG	No	А	3	++
6	ENIG	No	А	6	++
7	ENIG	Yes	А	3	++
8	ENIG	Yes	А	6	++
17	ENIG	No	В	3	++
18	ENIG	No	В	6	++
19	ENIG	Yes	В	3	++
20	ENIG	Yes	В	6	++
29	ENIG	No	C	3	++
30	ENIG	No	C	6	++
31	ENIG	Yes	C	3	++
32	ENIG	Yes	C	6	++
41	ENIG	No	D	3	++
42	ENIG	No	D	6	++
43	ENIG	Yes	D	3	++
44	ENIG	Yes	D	6	++

Table 10. Adhesion Test Results – ENIG Plating

++: No effect

+: Slight effect

0: Plating removed in some areas

-: Plating removed in most areas

Regardless of reflow, none of the cleaning agents impacted any of the surface finishes.

Results – Surface Test

The nickel and copper test were conducted on Board 2 from all trials for each surface finish as follows:

- Immersion Silver : Copper Test
- Immersion Tin : Copper Test
- ENIG : Nickel Test

Results of these tests were compared against the baseline values.

Results Copper Test - Immersion Silver

Baseline values for the amount of exposed copper were found to be 30 ppm for each board. Reference Figures 13A and 13B.



Figure 13A. Copper Test Result - Baseline Value: No Reflow



Figure 13B. Copper Test Result – Baseline Value: Lead-free Reflow

Results of the copper test conducted on Immersion Silver boards are detailed in Table 11.

Table 11. Copper Test Results - ImAg Plating

Trial #	Surface Finish	Reflow	Cleaning Agent	# Wash Passes	Copper Test Results
1	ImAg	No	А	3	100 ppm
2	ImAg	No	А	6	100 ppm
3	ImAg	Yes	А	3	100 ppm
4	ImAg	Yes	А	6	100 ppm
13	ImAg	No	В	3	30 ppm

14	ImAg	No	В	6	30 ppm
15	ImAg	Yes	В	3	20 ppm
16	ImAg	Yes	В	6	20 ppm
25	ImAg	No	С	3	10 ppm
26	ImAg	No	С	6	10 ppm
27	ImAg	Yes	С	3	0 ppm
28	ImAg	Yes	С	6	10 ppm
37	ImAg	No	D	3	10 ppm
38	ImAg	No	D	6	10 ppm
39	ImAg	Yes	D	3	10 ppm
40	ImAg	Yes	D	6	10 ppm

The baseline test results yielded an exposed copper level of 30 ppm regardless of whether the substrate was exposed to reflow. Thus, the authors inferred that the unprocessed boards themselves had a level of exposed copper prior to the wash process.

Additionally, it can be inferred from the results in Table 11:

- Cleaning Agent C (dynamic surfactant, inhibited) and Cleaning Agent D (pH neutral, micro phase inhibited) seemed to reduce the amount of exposed copper. This could be due to the inhibitors present in these formulations that protect the copper surface. Reference Figures 14A and 14B.
- Cleaning Agent A (dynamic surfactant, uninhibited) affected the Immersion Silver plating. The high copper values observed on the boards cleaned using this cleaning agent corroborated the visual inspection results. Reference Table 5 and Figures 14C and 14D.



Figure 14A. Copper Test, Cleaning Agent C – Trial 26: No Reflow, 6 passes



Figure 14B. Copper Test, Cleaning Agent D – Trial 38: Reflow, 6 passes



Figure 14C. Copper Test, Cleaning Agent A – Trial 2: No Reflow, 6 passes



Figure 14D. Copper Test, Cleaning Agent A – Trial 4: Reflow, 6 Passes

Results Copper Test - Immersion Tin

Baseline values for the amount of exposed copper were found to be 0 ppm for each board. Reference Figures 15A and 15B.



Figure 15A. Copper Test Result - No Reflow



Figure 15B. Copper Test Result - Lead-free Reflow

Results of the copper test conducted on Immersion Tin boards are detailed in Table 12.

Table 12	2 Conne	r Test]	Results	on	ImSn	Plating
	2. Coppe	I I CSU	Results	UII .	mon	1 lating

Trial #	Surface Finish	Reflow	Cleaning Agent	# Wash Passes	Copper Test Results
9	ImSn	No	А	3	0 ppm
10	ImSn	No	А	6	0 ppm
11	ImSn	Yes	А	3	0 ppm
12	ImSn	Yes	А	6	0 ppm
21	ImSn	No	В	3	0 ppm
22	ImSn	No	В	6	0 ppm
23	ImSn	Yes	В	3	0 ppm
24	ImSn	Yes	В	6	0 ppm
33	ImSn	No	С	3	0 ppm
34	ImSn	No	С	6	0 ppm
35	ImSn	Yes	С	3	0 ppm
36	ImSn	Yes	С	6	0 ppm
45	ImSn	No	D	3	0 ppm
46	ImSn	No	D	6	0 ppm
47	ImSn	Yes	D	3	0 ppm
48	ImSn	Yes	D	6	0 ppm

On the basis of the results in Table 12, the authors inferred that none of the cleaning agents had any compatibility issues with the Immersion Tin plating resulting in exposed copper. Reference Figures 16A and 16B for representative pictures with Cleaning Agent A.



Figure 16A. Copper Test, Cleaning Agent A – Trial 2: No Reflow, 6 passes



Figure 16B. Copper Test, Cleaning Agent A – Trial 4: Reflow, 6 Passes

Results Nickel Test – Electroless Nickel Immersion Gold

Baseline values for the amount of exposed nickel were found to be 5 ppm for each board. Reference Figures 17A and 17B.



Figure 17A. Nickel Test Result - No Reflow



Figure 17B. Nickel Test Result – Lead-free Reflow

Results of the nickel test conducted on Electroless Nickel Immersion Gold boards are detailed in Table 13.

Table 13.	Nickel	Test	Results	on	ENIG	Plating
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Trial #	Surface Finish	Reflow	Cleaning Agent	# Wash Passes	Nickel Test Results
5	ENIG	No	А	3	5 ppm
6	ENIG	No	А	6	5 ppm
7	ENIG	Yes	А	3	0 ppm
8	ENIG	Yes	А	6	0 ppm
17	ENIG	No	В	3	0 ppm
18	ENIG	No	В	6	5 ppm

19	ENIG	Yes	В	3	0 ppm
20	ENIG	Yes	В	6	0 ppm
29	ENIG	No	С	3	0 ppm
30	ENIG	No	С	6	0 ppm
31	ENIG	Yes	С	3	0 ppm
32	ENIG	Yes	С	6	0 ppm
41	ENIG	No	D	3	5 ppm
42	ENIG	No	D	6	5 ppm
43	ENIG	Yes	D	3	0 ppm
44	ENIG	Yes	D	6	0 ppm

On the basis of the results in Table 13, the authors inferred that none of the cleaning agents had any compatibility issues with the ENIG plating resulting in exposed nickel. Reference Figures 18A and 18B for representative pictures with Cleaning Agent A.



Figure 18A. Nickel Test, Cleaning Agent A - Trial 2: No Reflow, 6 passes



Figure 18B. Nickel Test, Cleaning Agent A - Trial 4: Reflow, 6 passes

Results – XRF Test

All XRF tests were conducted by an independent lab utilizing a Veeco Model XRF5100L with a 12 mil collimator.

All test results were compared with the control boards and the percent change in the thickness was calculated. A change of $\pm 5\%$ was considered within the scope of the experiment and those results are listed as "Negligible" and represented by "--" in Tables 14 – 16. The results of the XRF tests are detailed in Tables 14 – 16.

Trial #	Surface	Deflow	Cleaning	# Wash	XRF Test
1 mai #	Finish	Renow	Agent	Passes	Results
1	ImAg	No	А	3	
2	ImAg	No	А	6	-6.27%
3	ImAg	Yes	А	3	
4	ImAg	Yes	А	6	-5.50%
13	ImAg	No	В	3	
14	ImAg	No	В	6	
15	ImAg	Yes	В	3	
16	ImAg	Yes	В	6	
25	ImAg	No	С	3	
26	ImAg	No	С	6	
27	ImAg	Yes	С	3	
28	ImAg	Yes	С	6	
37	ImAg	No	D	3	
38	ImAg	No	D	6	
39	ImAg	Yes	D	3	
40	ImAg	Yes	D	6	

Table 14. Percent Change in Plating Thickness of Ag in ImAg Plating

--: Negligible

Table 15. % Change in Plating Thickness of Sn in Immersion Tin Plating

Trial #	Surface	Paflow	Cleaning	# Wash	XRF Test
111al #	Finish	Kellow	Agent	Passes	Results
9	ImSn	No	А	3	
10	ImSn	No	А	6	
11	ImSn	Yes	А	3	
12	ImSn	Yes	А	6	
21	ImSn	No	В	3	
22	ImSn	No	В	6	
23	ImSn	Yes	В	3	
24	ImSn	Yes	В	6	
33	ImSn	No	С	3	
34	ImSn	No	С	6	
35	ImSn	Yes	С	3	
36	ImSn	Yes	С	6	
45	ImSn	No	D	3	
46	ImSn	No	D	6	
47	ImSn	Yes	D	3	
48	ImSn	Yes	D	6	

--: Negligible

Table 16. % Change in Plating Thickness of Au and Ni inENIG Plating

Trial #	Surface Finish	Reflow	Cleaning Agent	# Wash Passes	XRF Test Results: Au Thickness	XRF Test Results: Ni Thickness	
5	ENIG	No	Α	3			
6	ENIG	No	Α	6			
7	ENIG	Yes	Α	3			
8	ENIG	Yes	Α	6			
17	ENIG	No	В	3			
18	ENIG	No	В	6			
19	ENIG	Yes	В	3			
20	ENIG	Yes	В	6			
29	ENIG	No	С	3			
30	ENIG	No	С	6			
31	ENIG	Yes	C	3			
32	ENIG	Yes	С	6			

41	ENIG	No	D	3	
42	ENIG	No	D	6	
43	ENIG	Yes	D	3	
44	ENIG	Yes	D	6	

--: Negligible

CONCLUSIONS

The potential impact of reflow and multiple passes through a spray-in-air inline cleaning process, utilizing engineered aqueous-based cleaning agents, on PCB substrates with Immersion Tin, Immersion Silver and ENIG surface finishes was evaluated through this study. Surface finish degradation was analyzed using visual inspection, adhesion test, and surface analysis. Surface analysis was conducted utilizing the copper test, nickel test, and XRF tests. Additionally, baseline values were determined through the surface analysis tests for use as a comparator to the substrates subjected to the process variables.

The results demonstrated that ENIG and ImSn finishes were robust and not degraded by reflow, the cleaning agent formulations or wash exposure time.

In the case of the ImAg finish, Cleaning Agent A (dynamic surfactant, uninhibited), was found to affect the plating material. The effect was particularly pronounced on the boards that were not reflowed after 6 passes through the spray-in-air inline cleaner, reference Trial 2 results. This was also verified by visual inspection (reference Table 5), copper tests (reference Table 11, Figures 14C and 14D) and XRF analysis (reference Table 14). It was also noted that the effect was more pronounced on larger pads. The authors theorized this is due to the fact that larger pads have a thinner plating layer than the smaller pads. Thus, plating layer thickness can also impact process compatibility.

Cleaning Agent B (micro phase, uninhibited), Cleaning Agent C (dynamic surfactant, inhibited) and Cleaning Agent D (pH neutral micro phase, inhibited) had no affect on the Immersion Silver plating. Reference Table 11. It was further noted that the inhibited cleaning agents, that is Cleaning Agent C and Cleaning Agent D, seemed to protect the exposed copper on the boards. This conclusion is derived from the results of the copper test wherein the boards cleaned using these cleaning agents showed a lower concentration of exposed copper than unprocessed control boards (reference Figures 14A and 14B).

Finally, exposure of the boards to the atmosphere for a period up to three weeks yielded no change in the degradation of the coating for all trials.

References

[1] J. Patel, U.Tosun, M.McCutchen, ZESTRON Americas "Comparative Cleaning Study to Showcase the Effective Removal of OA Flux Residues", <u>Proceedings of SMTA</u> <u>International</u>, October 2012.

[2] EMD Millipore Corporation; MQuant Copper Test

[3] EMD Millipore Corporation; MQuant Nickel Test

Appendix

Table 2.

Recommended Lead-free Reflow Oven Settings (°C)											
	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Zone 8	Zone 9	Zone 10	Cooling
Тор	100	120	150	180	190	200	210	230	245	255	3 Zones
Bottom	100	120	150	180	190	200	210	230	245	255	3 Zones
	Fan Speed at 50%Fan Speed at 60%										

Reflow Profile:

