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Testing and Analysis of Surface Mounted Lead Free Soldering Materials and Processes

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Abstract

The Massachusetts Toxics Use Reduction Institute (TURI) has sponsored a consortium of Massachusetts based corporations to investigate lead-free (Pb-free) surface mount soldering technology. The current effort is a Design of Experiments (DOE) analysis using three NEMI recommended tin silver copper (SnAgCu) alloys from three different solder manufacturers, five Pb-free PWB surface finishes, and two reflow environments. The consortium designed a special test PWB and that was used in the experiments. A modified visual test procedure was developed and the results are presented based on statistical analysis. Test PWBs with BGAs, leaded and chip components will be subjected to thermal cycling, and then tested for mechanical degradation. Standard tin-lead (SnPb) eutectic solder 63/37 reflow samples were used as a control.

Components on these test substrates include plastic and ceramic leaded SOICs, FPQFPs, an LCC, 45mm square ball grid arrays (BGAs) and small passive devices. This paper will discuss results along with the issues associated with each lead and PWB finish. Conclusions from this paper can be used as a guide to future product offerings, and to proper testing, reporting methods and recommendations to satisfy future Pb-free requirements.

Introduction

Currently, a large number of “toxic” materials used in electronics manufacture are under scrutiny. Legislation is in place or being considered to restrict, limit, or outright ban the use of Pb (lead) and brominated polymer flame-retardants used in electronics as well as other materials. Japan has implemented Pb-Free Electronics Guidelines and the European Economic Union is considering take-back and removal (WEEE – Waste Electrical and Electronic Equipment[1]) or outright banning import (RoHS – Restriction of Hazardous Substances[2]) while the

US EPA is working on TRI (Toxics Release Inventory[3]) reporting of hazardous components.

Massachusetts has founded the Toxics Use Reduction Institute (TURI) to assist companies in reducing their use of toxic substances. They have funded a number of university led programs including Pb-free electronics assembly development. To increase the scope of this effort, the University of Massachusetts – Lowell, has formed a consortium of local industries to help develop Pb-free electronics. Corporate members are: Air Products and Chemicals, Analog Devices, BTU International, M/A-COM, Raytheon, Sanmina-SCI, Schneider Electric-Automation Business, and Texas Instruments.

Several studies (DOEs) have already been published[4-7]. The effort this year is focusing on solder composition, PWB finishes (solderability preservatives), and lead finishes.

Experimental Design Matrix

A designed experiment was developed with the following variables:

1. PWB Finishes – Five Treatments– Solder Mask Over Bare Copper with Hot Air Solder Leveling (SMOBC/HASL), Matte Finish Tin (Sn) Electroplate, Immersion Silver (Ag), Organic Solder Preservative (OSP), and Electroless Nickel Immersion Gold (ENIG).
2. Reflow Atmospheres – Two Treatments – Air and Nitrogen.
3. Solder Pastes – Three Treatments – all with the same alloy composition – 95.5Sn-3.8 Ag-0.7Cu (NEMI recommended) from three different vendors, all incorporating no-clean fluxes.
4. Component Lead Finishes – Four Treatments – “Whisker-free” Sn, Tin/Silver/Copper, Nickel/Palladium/Gold, and Nickel/Gold.
5. Sn-Pb eutectic solder PWB using the solder treatments as control PWBs.

The Design matrix is shown in Table 1.

Table 1. Experimental Design Matrix

Experimentation Matrix				
				(Two (2) Circuit Boards per Trial)
PWB	**PWB Finish**	**Solder paste**	***Reflow Atmosphere**	**Component Finish**
1	SMOBC/HASL	“A”	Air	Pb- free
2	SMOBC/HASL	“A”	Nitrogen	Pb- free
3	SMOBC/HASL	“B”	Air	Pb- free
4	SMOBC/HASL	“B”	Nitrogen	Pb- free
5	SMOBC/HASL	“C”	Air	Pb- free
6	SMOBC/HASL	“C”	Nitrogen	Pb- free
7	SMOBC/HASL	Standard Sn-Pb	Air	Sn-Pb Leads
8	OSP	“A”	Air	Pb- free
9	OSP	“A”	Nitrogen	Pb- free
10	OSP	“B”	Air	Pb- free
11	OSP	“B”	Nitrogen	Pb- free
12	OSP	“C”	Air	Pb- free
13	OSP	“C”	Nitrogen	Pb- free
14	OSP	Standard Sn-Pb	Air	Sn-Pb Leads
15	ENIG	“A”	Air	Pb- free
16	ENIG	“A”	Nitrogen	Pb- free
17	ENIG	“B”	Air	Pb- free

18	ENIG	“B”	Nitrogen	Pb- free
19	ENIG	“C”	Air	Pb- free
20	ENIG	“C”	Nitrogen	Pb- free
21	ENIG	Standard Sn-Pb	Air	Sn-Pb Leads
22	Matte Sn	“A”	Air	Pb- free
23	Matte Sn	“A”	Nitrogen	Pb- free
24	Matte Sn	“B”	Air	Pb- free
25	Matte Sn	“B”	Nitrogen	Pb- free
26	Matte Sn	“C”	Air	Pb- free
27	Matte Sn	“C”	Nitrogen	Pb- free
28	Matte Sn	Standard Sn-Pb	Air	Sn-Pb Leads
29	Ag	“A”	Air	Pb- free
30	Ag	“A”	Nitrogen	Pb- free
31	Ag	“B”	Air	Pb- free
32	Ag	“B”	Nitrogen	Pb- free
33	Ag	“C”	Air	Pb- free
34	Ag	“C”	Nitrogen	Pb- free
35	Ag	Standard Sn-Pb	Air	Sn-Pb Leads

*Nitrogen was supplied by Air Products and Chemical and contained 50 ppm Oxygen for these experiments.

As seen, all test PWBs were run versus the standard 63Sn-37Pb eutectic, no-clean solder as a control. Package types included several SOICs, a FPQFP, an MLP, a BGA, a ceramic SOIC, and chip capacitors and resistors.

The test PWB was laid out at M/A-COM taking into account daisy chain resistance test capabilities in some of the parts and fabricated by Sanmina-SCI with the five different finishes. Pastes were obtained from three vendors and a reflow profile was developed based on the manufacturers’ product data sheets. A reflow profile board was populated with parts and three K-probe thermocouples (TC) were attached to the surface. One TC was attached at the leading edge of the PWB, one at the lead attach area of a large QFP and one near the trailing edge. The thermocouples were connected to an industry standard data logger. The thermal readings were downloaded to the data collector software for comparison to the manufacturer recommended profiles. All three manufacturers recommended a 'ramp to spike' curve. Several runs were performed to ensure consistent performance. The reflow profile used for all three Pb-free solders is shown in Figure 1. Solder paste prints were made using a 0.006” thick stainless steel laser cut, electropolished stencil. Ten percent aperture reductions were used on the fine pitch devices. PWBs were assembled at Schneider Electric on their assembly line consisting of an MPM AP-25 screen printer, Siemens S20 and F5 placement equipment and a BTU Pyramax 98N Reflow Oven with Air and Nitrogen capability supplied by BTU International for this experiment. The Schneider plant maintains a Relative Humidity (RH) level between 35-40%.

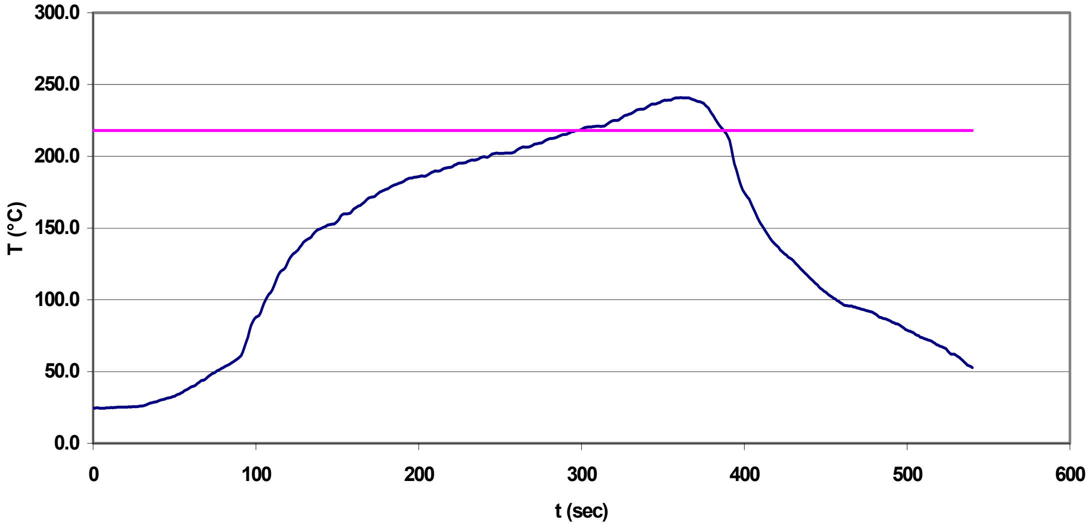

Figure 1. Reflow Profile for SMT Board Assembly

After reflow, boards were packaged in ESD bags and taken to M/A-COM where two University of Massachusetts – Lowell senior students visually inspected the solder joints based on training by a certified IPC inspector / trainer. Inspection criteria were established as follows: Total Defects, Cold Solder joints, Non-wetting, Solder Balls, Dewetting, Bridging, Pinholes, Shiny Appearance, Smooth Appearance, and Flux Residue. X-ray radiography of the BGA solder joints was also performed. Initial inspection data has been tabulated and statistically analyzed by University of Massachusetts – Lowell and Air Products. To date Board Finish, Atmosphere, and paste (A, B, C) have been analyzed. Work is in progress on lead finish and component type.

Results

Assembly Process

The major difficulties encountered in assembly were with stencil printing and placement system vision. In spite of using print parameters close to those in the application notes supplied for the three pastes, paste A had a tendency to adhere to the sides of the stencil openings. This resulted in scant prints on some of the fine pitch apertures. Paste B clogged the necessitating cleaning after every four or five prints. Paste C performed as expected with little difficulty. All three pastes exhibited good tack or component holding qualities during and after placement. Vision problems were associated with the Sn-Pb version of the BGA. The difference in appearance (reflectivity) of the Sn-Pb spheres caused the vision system (programmed for the Pb-free spheres) to reject many of the Sn-Pb BGAs. These had to be placed by hand which may affect some results.

Visual Defects - Statistics

Eight main categories of common defects were selected and all boards were inspected. Those defects observed were photographed and recorded into a spreadsheet. After statistical analysis the following significant effects were determined (all shown by the letter Y and a number indicating the rank starting with 1 being the most significant):

Table 2: Statistically Significant Effects Summary

	Main Effects			2 Factor Interactions			3 Factor Interaction
Property	Finish	Paste	Atmosphere	Finish*Paste	Finish*Atmosphere	Paste*Atmosphere	Finish*Paste*Atmosphere
Total Defects	Y - 3	Y - 2	Y - 1	N	N	Y	N
Cold Solder Joining	Y - 3	Y - 2	Y - 1	N	N	Y	N
Nonwetting	N	Y - 2	Y - 1	N	N	N	N
Solder Balls	Y	N	N	N	N	N	N
Dewetting	N	N	N	N	N	N	N
Bridging	Y	N	N	N	N	N	N
Pin/Blow Holes*	N	N	N	N	N	N	N
Shiny**	Y - 1	Y - 2	N	N	N	N	N
Residue**	N	Y - 1	Y - 2	N	N	N	N
Smooth**	N	N	N	N	N	N	N

Table 3: Atmosphere Results Summary

	Atmosphere	
Property	Air	Nitrogen
Total Defects	**150.30**	**6.00**
Cold Solder Joining	**102.70**	**1.10**
Nonwetting	**33.30**	**1.60**
Solder Balls	9.07	2.83
Dewetting	5.10	0.43
Bridging	0.13	0.03
Pin/Blow Holes*	0.00	0.00
Shiny**	0.63	0.43
Residue**	**0.83**	**0.47**
Smooth**	0.80	0.97

Nitrogen reflow atmosphere was shown to be significant in total defects, cold solder joints, non-wetting and residue generated.

Table 4: Pb-Free Paste Results Summary

	Pb-Free Paste		
Property	B	A	C
Total Defects	**11.35**	**171.25**	**51.90**
Cold Solder Joining	**0.20**	**137.45**	**18.10**
Nonwetting	**2.60**	**20.25**	**29.50**
Solder Balls	8.00	6.35	3.50
Dewetting	0.50	7.15	0.65
Bridging	0.05	0.05	0.15
Pin/Blow Holes*	0.00	0.00	0.00
Shiny**	**0.80**	**0.30**	**0.50**
Residue**	**0.85**	**0.70**	**0.40**
Smooth**	0.95	0.90	0.80

A statistically significant Paste effect means that one or more of the pastes studied differ significantly from each other in terms of the type and / or number of defects generated. Significance is noted with bold numbers

Table 5: Board Surface Finish Results Summary

Property	Finish				
	ENIG	Imm. AG	Matte Sn	OSP	SMOBC/HASL
Total Defects	**20.42**	**29.67**	**62.92**	**107.58**	**170.25**
Cold Solder Joining	**5.83**	**13.83**	**43.75**	**101.75**	**94.42**
Nonwetting	7.75	13.42	6.50	3.50	56.08
Solder Balls	**6.08**	**2.00**	**1.33**	**1.67**	**18.67**
Dewetting	0.75	0.42	11.33	0.67	0.67
Bridging	**0.00**	**0.00**	**0.00**	**0.00**	**0.42**
Pin/Blow Holes*	0.00	0.00	0.00	0.00	0.00
Shiny**	**0.17**	**0.75**	**0.50**	**0.67**	**0.58**
Residue**	0.83	0.58	0.42	0.67	0.75
Smooth**	0.83	0.92	0.75	0.92	1.00

A statistically significant Board Finish effect means that one or more of the surface finishes studied differ significantly from each other. Significance is noted with bold numbers

Legend for all Tables

* Pin/Blow Holes: No defects of this type were observed
** Qualitatively measured properties
Statistically significant effects are bolded and in blue.

As seen in Table 2, the two major defects observed were cold solder joints and non-wetting. All major variables impacted these two defects and a significant interaction effect between paste and atmosphere on cold solder joints was also observed. Solder ball defects were also impacted by board finish. Some variables also had a significant effect on the solder joint appearance with respect to shininess and flux residue. Since Pb-free solder is different in appearance, these were considered important as well.

Visual Defects - Images

Following (Figures 2-10) are some representative photographs of several solder joint visual defects and appearance issues on various package types:

Figure 2. At the left, the expected higher wetting angles for Pb-free solder joints are observed. At the right, non-coalesced paste spheres are seen in this Air reflow sample Paste A on SMOBC – HASL.

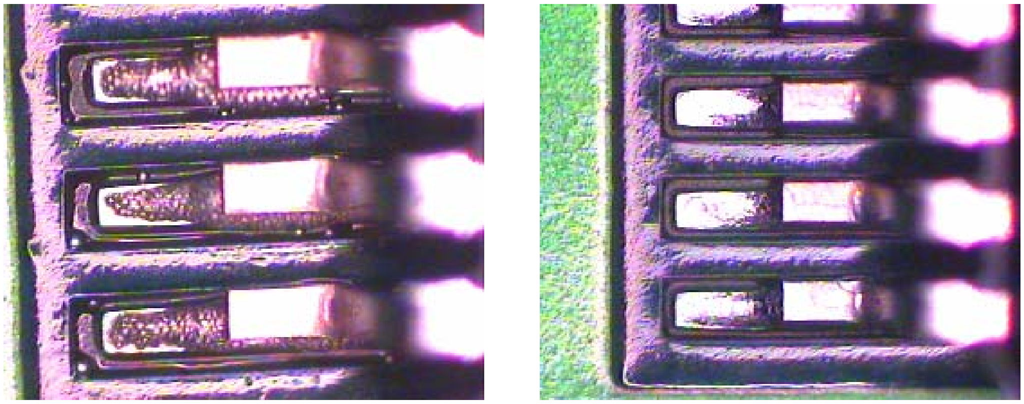

Figure 3. Left: Paste A reflowed in Air – Right: Paste A in Nitrogen - fine pitch print, Ni-Pd-Au lead – SMOBC-HASL.

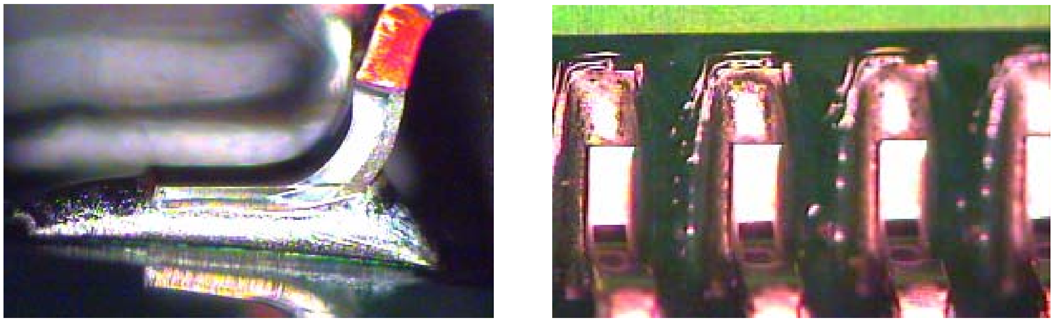

Figure 4. Paste A in Air, SOIC leads on the same board. Left: lead finish plated tin - Right: Ni-Pd-Au plated leads –SMOBC-HASL

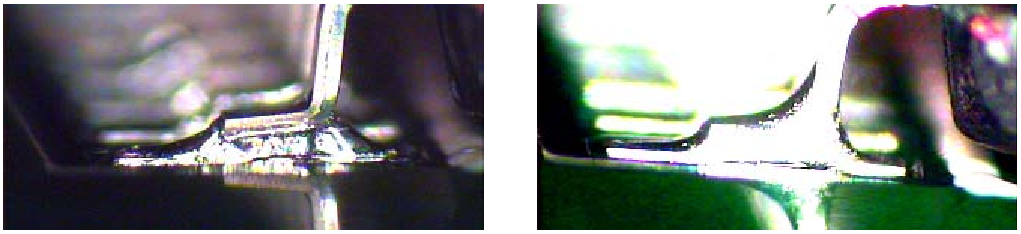

Figure 5. Paste B in Air – left; Paste B in Nitrogen – right – SMOBC-HASL.

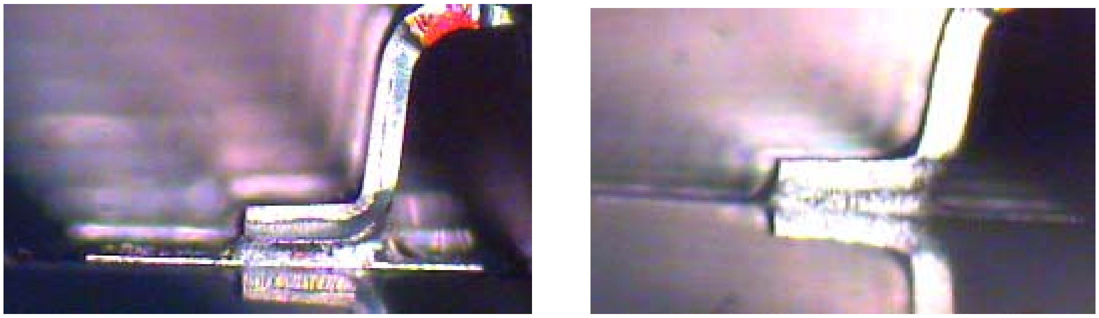

Figure 6. Paste C in Air – left; Paste C in Nitrogen – right – SMOBC-HASL.

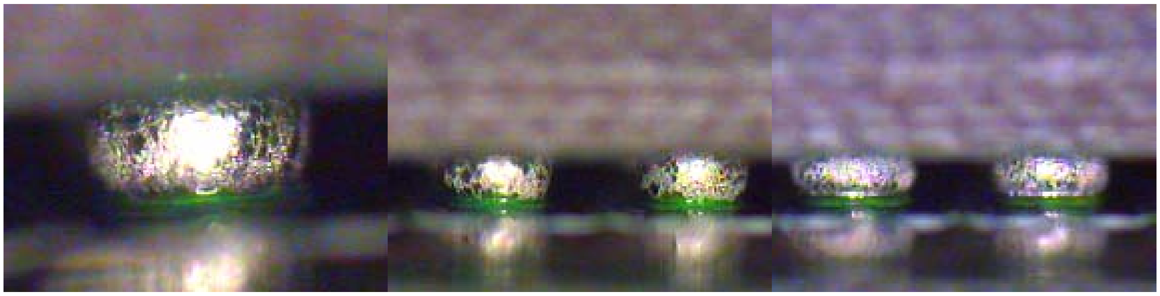

Figure 7. BGA solder joints – Left – Paste A in Air – SMOBC-HASL – Center – Paste B in Nitrogen – ENIG – Right – Paste C in Air - ENIG

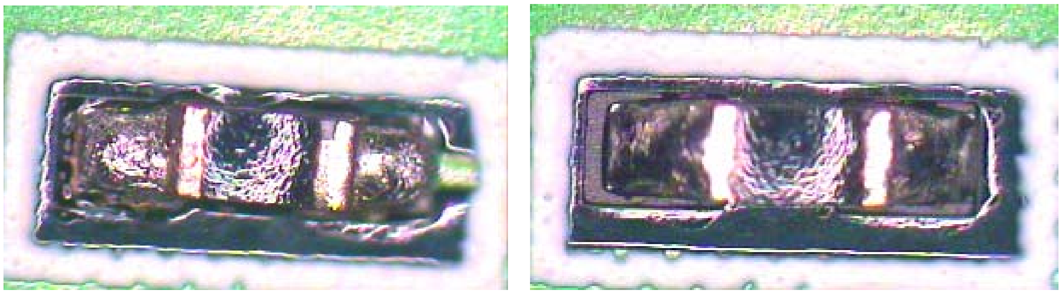

Figure 8. Pb-free (Sn) resistors – Paste C on immersion silver, left in Air, right, in Nitrogen.

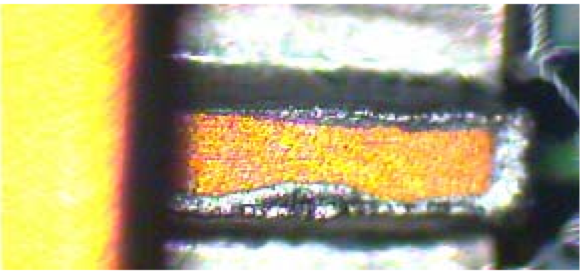

Figure 9. Paste B in Nitrogen on SMOBC-HASL – gold lead finish.

Figure 10 – Pb-Sn reflow control – Sn leads on matte tin finish.

Other experimental results to be reported later include: 1) BGA sphere attach experiments, 2) x-ray analysis for voids and BGA bridging, 3) oxygen content of reflow Nitrogen effects, 4) effects of reflow profile, 5) and pull and shear test data before and after thermal cycling.

Discussion

Visual inspection, of course, does not tell the entire story. It is a good method of finding many defects but does not reveal weak solder joints, large internal voids, or many BGA solder joint defects. Moreover, the correlation between appearance and reliability has not been established for Pb-free solders, in the way that it has for Sn63 – type solders. This correlation will be explored during the continuation of this study.

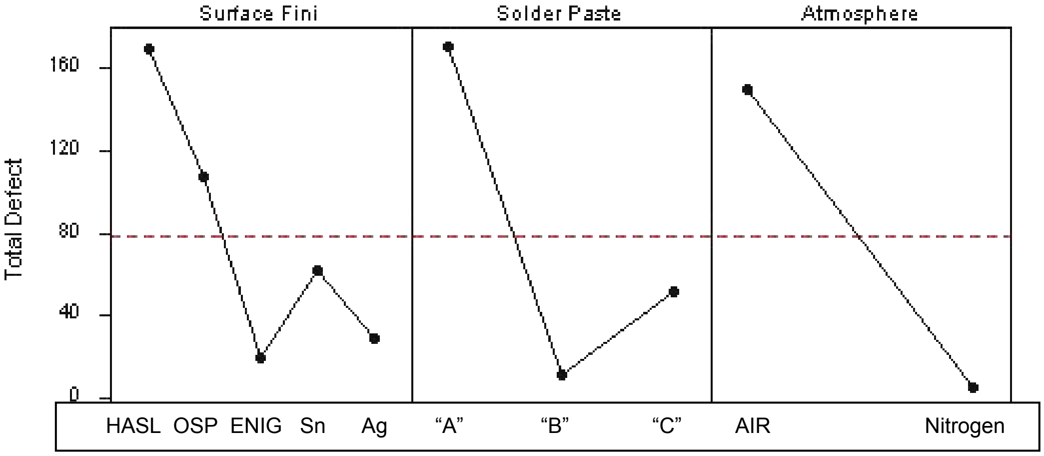

Figure 11. Defect Density versus Independent Variables

Based on Figure 11, ENIG, Paste “B”, and Nitrogen would be considered “winners” with Ag a close second for board finish. Within the confines of effect of these variables on visual defects / appearance issues this is correct. However, manufacturing cost issues were not addressed as part of the overall analysis. Nitrogen does cost more than using ambient Air so defect costs may have to be offset by Nitrogen costs. Electroless nickel used in ENIG has often been associated with “black pad” solderability defects (not an issue in this study) that may occur in some circuit card assemblies. As noted above, paste B clogged the stencil requiring cleaning after every four or five prints and thus could have a large effect on throughput and other line costs.

Consider Figure 3 above. Paste A clung to the aperture walls during printing resulting in scant prints on fine pitch pads. The flux tended to flow during reflow ramp and soak and so, with an inappropriate volume of paste, was not able to prevent the paste spheres from re-oxidizing before reflow. The paste under the lead did wet and flow, as did areas with more paste volume. Perhaps an experiment to develop a better stencil print would improve this result. The other alternative, Nitrogen, is more forgiving so this paste may be fine for larger pitch or when using Nitrogen for a reflow atmosphere.

Returning to Figure 11, paste “C” had more defects, mainly in Air, but also gave a good print nearly every board. Experiments may improve performance for all pastes, "C", "B", and "A". One must also consider that these pastes are not necessarily fully developed with good histories as Pb-free processing itself is still in its infancy.

Figures 4 – 8 all show solder joints with "acceptable" appearances if one considers the higher wetting angle associated with Pb-free pastes. Figure 9 shows the effect of a thick gold finish on reflow. The results are similar to what one would find in a eutectic Sn-Pb solder joint. The gold, as it dissolves in the molten solder, raises the melting temperature in the limited solder volume available to the point when further wetting is "frozen out" leaving a wave front as shown.

Total Defect analysis results

Table 6 - Statistical Analysis – Total Defects

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                        ANOVA for 0.35 Power Transformed Total Defect Data

                                       The GLM Procedure

Dependent Variable: Total Defect
                                            Sum of
       Source                      DF      Squares     Mean Square    F Value    Pr > F

       Model                       29    363.4838458    12.5339257       8.23    <.0001
       Error                       30     45.7076735     1.5235891
       Corrected Total             59    409.1915194

                       R-Square     Coeff Var      Root MSE       TD Mean
                       0.888298      43.38638      1.234338      2.844989

       Source                    DF      Type III SS     Mean Square    F Value    Pr > F

       Board Finish               4      44.6816022      11.1704005       7.33    0.0003
       Paste                      2      78.9621665      39.4810833      25.91    <.0001
       Atmosphere                 1     132.3624551     132.3624551      86.88    <.0001
       Finish*Paste               8      16.0395976       2.0049497       1.32    0.2735
       Finish*Atmosphere          4      15.2827444       3.8206861       2.51    0.0629
       Paste*Atmosphere           2      54.3289039      27.1644519      17.83    <.0001
       Finish*Paste*Atmosphere    8      21.8263762       2.7282970       1.79    0.1184
```

As seen above, the ANOVA (Analysis of Variance) is significant for the overall experiment and for the variables highlighted with probabilities Pr less then .05.

Ryan-Einot-Gabriel-Welsch Multiple Range Test for Total Defects
(Board Finish)

Means with the same REGWQ Grouping letter (A, B) are not significantly different.

REGWQ Groupings	Mean	N	Finish
A	170.25	12	SMOBC/HASL
B	107.58	12	OSP
B			
B	62.92	12	Matte Sn
B			
B	29.67	12	Imm. AG
B			
B	20.42	12	ENIG

Interpretation: The Board Finish level SMOBC/HASL significantly differs from all other finishes. No other finishes were found to be statistically different from one another at the 0.05 level.

Ryan-Einot-Gabriel-Welsch Multiple Range Test for Total Defects (Paste)

Means with the same REGWQ Grouping letter (A, B, …) are not significantly different.

REGWQ Grouping	Mean	N	Paste
A	171.25	20	A Pb-Free
B	51.90	20	C Pb-Free
C	11.35	20	B Pb-Free

Interpretation: All Pastes were found to differ significantly from all other pastes. B Pb-Free performed best.

Ryan-Einot-Gabriel-Welsch Multiple Range Test for Total Defects (Atmosphere)

Means with the same REGWQ Grouping letter (A, B, …) are not significantly different.

REGWQ Grouping	Mean	N	Atmosphere
A	150.33	30	Air
B	6.00	30	Nitrogen

Interpretation: Nitrogen preformed significantly better than Air.

Ryan-Einot-Gabriel-Welsch Multiple Range Test for Total Defects (Paste x Atmosphere Interaction)

Means with the same REGWQ Grouping letter (A, B) are not significantly different.

REGWQ Grouping	Mean	N	Paste x Atmosphere Interaction
A	337.10	10	A Pb-Free with Air
B	98.70	10	C Pb-Free with Air
C	15.20	10	B Pb-Free with Air
C			
C	7.50	10	B Pb-Free with Nitrogen
C			
C	5.40	10	A Pb-Free with Nitrogen
C			
C	5.10	10	C Pb-Free with Nitrogen

Interpretation: The A Pb-Free, Air combination was significantly worse than all other combinations. The C Pb-Free, Air combination was significantly worse than all other remaining combinations. The bottom four combinations could not be told statistically apart from each other within the limitations of the current study.

Only in the case of solder paste B; it was shown that there is no significant difference between the use of Air or Nitrogen. However, as noted earlier this paste exhibited certain process issues relating to the cost of more frequently cleaning the stencil in the production process.

Part of this effort is to develop inspection criteria for Pb-free solder joints. Two items inspected for were shiny appearance and flux residue. Like other effects these may bear on each other. Flux residue is often glossy and may cause a joint to "look better" than a dry joint. This may be further confused with Air versus Nitrogen, as a joint with residue may have less of a tendency to oxidize while cooling than an unprotected surface.

As noted, there is considerable work to be done in terms of mechanical properties (lead pull, ball and chip shear), joint x-rays for voids and BGA flaws, and stress testing via thermal cycling (0 to 100 °C) followed by more mechanical testing. Further analysis on lead finishes also is in progress and requires this data.

Conclusions to Date

We have shown the effects of atmosphere, paste selection, and surface finish on visual appearance defects. While Nitrogen and paste "B" yielded the fewest defects and SMOBC – HASL was significantly worse as a surface finish, the assembly process was not optimized for any of the variable options. Further, throughput and cost can be significant issues that may override some of these results. Other data to be taken needs to be correlated with these findings to further clarify the effects of these variables on solder joint reliability and yield.. These data show it is possible to obtain visually acceptable solder joints using a variety of board finishes, lead finishes, paste formulations and Air/Nitrogen combinations. Visual inspection is often the only means of defect detection other than circuit testing, and the two often verify one another thus indicating a good Pb-free reflow process with high yield can be achieved.

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- Tom Bzik of the Statistical Science Group**,** Air Products, for statistical analysis and discussions.

References and Consortium Publications

1. Lead Free Electronics Workshop, hosted by Lucent technologies, Merrimack Valley Works, North Andover. MA, Tuesday April 13, 2000.
2. TURI continuing education conference, "TUR in Massachusetts: the Next Generation" on Tuesday April 25th 2000, Session, C2, the Lead Free electronics, at the Best Western Royal Plaza and Trade Center, Marlborough, MA.
3. State of Massachusetts Legislative committee on education policy, UMASS President Bulger's Office, May 4th, 2000.
4. IMAPS New England, 27th annual symposium and exhibition, May 2000, Holiday Inn Conference Center, Boxborough, MA, session F, Surface Mount Technology.
5. CEAM /TURI Colloquy on University Research in Sustainable Technologies Program (URST) at UMass Lowell, June 2000
6. 2nd Workshop on Lead-Free Electronics, Technical Issues and Challenges in the Transition To Lead-Free Technologies, Thursday, June 29, 2000, at BTU International North Billerica, Massachusetts
7. Consortium authors, "Design Of Experiments For Lead Free Materials, Surface Finishes And Manufacturing Processes Of Printed Wiring Boards", published by the SMTA International Conference at Rosemount trade center, Chicago, IL, September 2000
8. Consortium authors, "Design Of Experiments For Lead Free Materials, Surface Finishes And Manufacturing Processes Of Printed Wiring Boards", published by the Chinese Electronics Association Journal
9. Consortium authors, "Selecting Material and Process Parameters for Lead Free SMT Soldering Using Design of Experiments Techniques", Apex Conference, January 2001, San Diego, CA,
10. Consortium authors, "Reliability Testing Techniques For Lead Free Soldering Of SMT Technology", ETRONIX Conference, Anaheim, CA, March 2001.
11. "Process and Material Selection for zero defects and superior adhesion Lead Free SMT soldering", SMTA International Conference, Chicago, IL September 2001.
12. Shina, S., <u>Six Sigma for Electronics Design and Manufacturing</u>, McGraw-Hill, NY, 2002.