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## Abstract

The purpose of this study was to determine the benefits of using inert atmospheres to improve the reflow soldering process for surface mount (SMT) printed circuit board assembly. With the push towards the European Union environmental legislation and initiatives that include the ROHS, WEEE and REACH as well as similar initiatives in Japan, China and Korea, the conversion to lead-free solder alloys has posed significant challenges for the electronics assembly community. The cost of this conversion has been high and the key for the global assembly community is how to process these SMT boards with similar quality and performance to those observed with the traditional lead-based solder alloys.

The conditions tested were oxygen levels and reflow temperatures in the reflow zone of a typical forced convection furnace. The effects observed were: (1) improved solder wetting, (2) reduced flux residue and ionic contamination, and (3) lower processing temperatures. The lead-free solder paste alloys used were from three different suppliers and had the same nominal alloy composition of 96.55% Sn / 3.0% Ag / 0.7% Cu. The printed circuit board designs were based on IPC standard designs and an internally designed board to measure solder wetting.

The results of this study demonstrated that nitrogen inert atmospheres, with O<sub>2</sub> ppm levels of 1000 ppm or lower have a positive effect in improving solder wetting, reduction in flux residue and lower processing temperatures.

## Introduction

With the push towards the European Union environmental legislation and initiatives that include the ROHS, WEEE and REACH as well as similar initiatives in Japan, China and Korea, the conversion to lead-free solder alloys has posed significant challenges for the electronics assembly community. The cost of this conversion has been high and the key for the global assembly community is how to process these SMT boards with similar quality and performance that was observed with the traditional lead-based solder alloys.

The key function of electronic packaging is to provide a communication link between the integrated circuit (IC) and the system as well as to protect the IC from mechanical and environmental damage. Integrated circuit line geometries are becoming smaller and with this the increasing speed of information transfer is placing challenging demands on the packaging and assembly industries due to achieve the shortest signal path between functional components. The trend towards miniaturization of systems from consumer electronics to mobile information systems has driven the electronics industry to develop innovative packaging and assembly process technologies to accommodate this trend. Electronics packaging and assembly processes must provide reliable systems at a relatively low cost per unit.

The purpose of this study was to determine the benefits of using an inert atmosphere (nitrogen) to improve the reflow soldering process using industry standard lead-free solder pastes for surface mount printed circuit board assemblies. There are several key factors that assembly houses must

recognize when using lead-free solder pastes. Standard lead-free (96.5% Sn / 3.0% Ag / 0.7% Cu) SAC alloy has a higher melting point than lead-based solder alloy. The melting point range for the SAC alloy is 217 to 221°C, with a reflow peak temperature of 240 to 260°C. With lead-free solder paste the flux activity is higher in order to effectively reduce tin oxide formation in an air atmosphere reflow process. In an air atmosphere reflow there is a greater opportunity to develop poor wetting, cold solder joints, and insufficient solder. Also in addition, the increase in flux residues reduces post-solder inspection accuracy in both visual and AOI methods.

This study was contracted by Air Products and Chemicals, Inc. (APCI) and conducted at the American Competitive Institute (ACI) in Philadelphia, PA, U.S.A. The conditions tested included:

1. Oxygen parts per million (ppm) levels in the reflow zone.
2. Reflow process temperature

The effects analyzed were:

1. Improved solder wetting
2. Reduced flux residue and ionic contamination
3. Lower reflow processing temperatures

### **Experimental Design**

The statistical design of experiments plays an important role in experimentation. To obtain meaningful information from experimentation, the variables under investigation must be varied in a systematic way. A well-structured design will be able to independently evaluate the effect of each variable on the measured response and reveal any interactions or synergistic effects that may be occurring between variables. Three industry standard SAC alloy solder pastes were used for this study. The solder pastes used were designated for this study as “S”, “A” and “I”. The printed circuit board designs that were used in this program were as follows:

1. Standard SMT configuration as designed by ACI (Fig. 1)
2. IPC B-36 board configuration for the ionic analysis (IA) and flux residue analysis (Fig. 2)
3. IPC B-24 board configuration for the surface insulation resistance analysis (SIR) (Fig. 3)
4. Wetting Test pattern configuration ( Fig. 4)

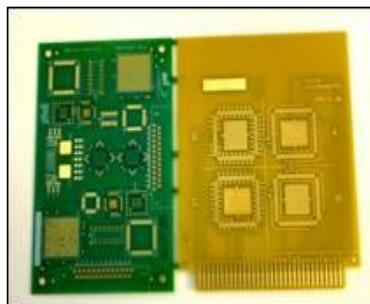


Figure 1. A standard SMT test board used in the study.

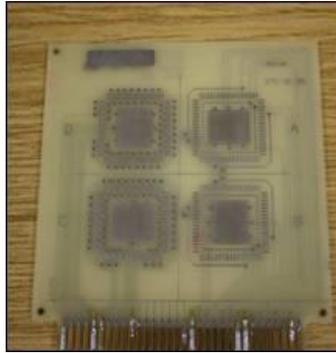


Figure 2. IPC B-36 board configuration for the ionic analysis (IA) and flux residue analysis

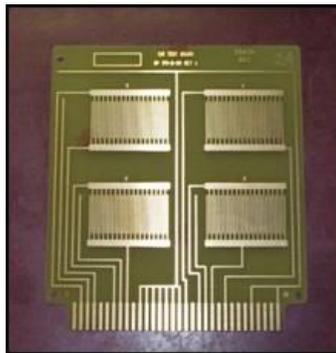


Figure 3. IPC B-24 board configuration for the surface insulation resistance analysis (SIR)

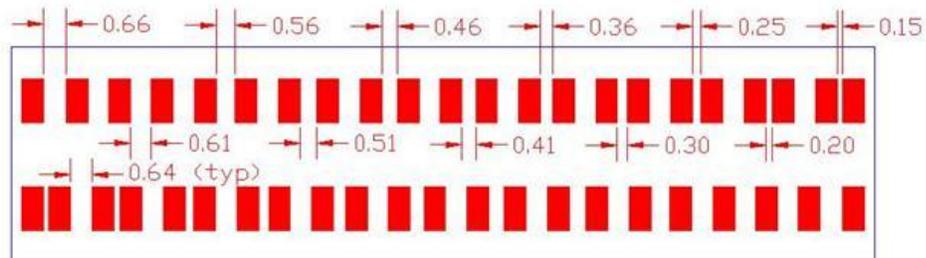


Figure 4. Wetting test pattern configuration

The SMT, IA and SIR boards' surface finish was electroless nickel and immersion gold (ENIG). For determining the effects of reflow conditions on the quality of solder joints, temperature, oxygen levels and solder paste chemistries were varied. Temperature was treated as a continuous variable, and varied from 230 to 250°C, including a mid-point temperature to test for curvature. Solder paste types were treated as categorical values. It was also necessary to treat oxygen as a categorical, rather than continuous variable. Oxygen levels tested were 100, 500, 1,000, 5,000 and 210,000 ppm, which are typical settings that could be used in the manufacturing process. A software developed in-house, Stat-Studio<sup>®</sup>, was used to construct the experimental designs and analyze the results.

The design of experiment for Phase I (IS, SIR and Flux residue) is shown in Table 1:

**TABLE 1. PHASE I DOE**

Run #	Temp	O <sub>2</sub> ppm	Solder Paste
1	240	210000	S
2	240	210000	S
3	235	210000	A
4	240	100	S
5	245	500	I
6	235	1000	S
7	235	5000	A
8	245	100	S
9	245	1000	A
10	245	210000	A
11	245	500	S
12	245	5000	S
13	235	1000	A
14	245	5000	I
15	245	5000	A
16	235	100	I
17	235	210000	I
18	245	100	I
19	235	100	A
20	245	210000	I
21	235	5000	S
22	240	100	A
23	240	1000	I
24	235	500	A
25	245	1000	S
26	235	500	S
27	235	500	I

The design of experiment for Phase II (Wetting Analysis I) is shown in Table 2:

**TABLE 2**

Reflow Temperature	O <sub>2</sub> ppm	Solder Paste
230	210,000	S
230	100	S
230	1,000	S
255	210,000	S
255	100	S
255	1,000	S

The design of experiment for Phase III (Wetting Analysis II) is shown in Table 3.

**TABLE 3**

Solder Paste	Reflow Temperature	O <sub>2</sub> ppm
A	230	100
A	230	1,000
A	230	2,000
A	230	5,000
A	230	210,000
A	245	100
A	245	1,000
A	245	2,000
A	245	5,000
A	245	210,000
A	260	100
A	260	1,000
A	260	2,000
A	260	5000
A	260	210000
S	230	100
S	230	1,000
S	230	2,000
S	230	50,00
S	230	210,000
S	245	100
S	245	1,000
S	245	2,000
S	245	5,000
S	245	210,000
S	260	100
S	260	1,000
S	260	2,000
S	260	5,000
A	260	210,000

## Experimental Procedures and Results

### **Phase I Program – Surface Insulation Resistance and Ionic Analysis**

Based on the design of experiment in Table 1, the following test procedure was run to determine the SIR and IA results.

The two-board panel was designed with the standard IPC B-36, unmasked SIR test board allowing for ion chromatography testing and SIR testing (Fig. 2). The B-36 board was populated with four dummy (no die) 68 I/O castellated gold plated leadless ceramic chip carriers (LCCs). The boards were cleaned prior to any assembly step. Twenty-seven assembled panels were tested along with one bare panel for both the IC and SIR tests.

#### Ion Chromatography Analysis and Instrumentation

The analysis was done in accordance with IPC TM-650 2.3.28 "Ionic Analysis of Circuit Boards, Ion Chromatography Test Method".

Dionex DX-500 High Performance Chromatograph using Peak Net<sup>®</sup> software version 6.20

Column: Ion Pac CG12A Guard column

Ion Pac CS12A Analytical column

Mobile phase: Dionex AS4A Eluent concentrate prod no. 39513 diluted 100x in deionized water.

Flow rate = 2.00 mls/min.

Printed circuit boards (PCB) were extracted with 75%/25% isopropyl alcohol/water using an 80°C water bath for 1 hour. The extracted solution was analyzed against known standards to confirm the presence of and quantify each anion in units of µg/ml. The surface area was determined and the final results reported in µg/inch<sup>2</sup>. ACl's maximum recommended amounts of fluoride, chloride, bromide, nitrate, and sulfate for bare boards are 2, 4, 5, 1 and 3 µg/in<sup>2</sup> respectively.

The recommended levels of ionic contamination for populated assemblies will depend upon the application. However, for typical component packages on FR-4 or a like substrate, the maximum recommended amounts of fluoride, chloride, bromide, nitrate, and sulfate are 2, 9, 15, 1 and 10 µg/in<sup>2</sup> respectively. Both sets of acceptance criteria were developed from experience and in conjunction with industry leaders.

#### Surface Insulation Resistance Testing

A B-24 board was tested for residue ionic contaminants as per IPC-TM-650 2.3.25 "Detection and Measurement of Ionizable Surface Contaminants by Resistivity of Solvent Extract (ROSE)" prior to any assembly work. It was incorporated in the testing and acted as a secondary control sample.

The B-36 boards unlike the B-24 board (Fig. 3) allow for components. However both have the similar comb pattern consisting of a bias voltage side and a measurement side. The four quadrants on the board are split into two regions of different test locations. The SIR testing was per IPC-TM-650-2.6.3.3 and involved connection of the comb patterns from each board to a SIRometer and placement of the wired boards into a heat/humidity chamber that was raised to 85°C and 85% relative humidity.

A test voltage of 100 volts DC was applied for one minute to each test point before a resistance test. This resistance test was done at 24, 96 and 168 hours. As a precaution, the SIRometer removes the power to particular traces that fall below the limit of  $1 \times 10^6$  Ohms. A resistance test was also done at 25°C and 50% relative humidity before and after the 85/85 test conditions. During the time period resistance measurements were not taken, a bias voltage of -50 volts DC was applied to all test points. At the conclusion of the test the LCCs were removed and examined under an optical microscope at a minimum of 10X.

J-STD-001D, appendix C requires that the average resistance results for the 96 and 168 hour results shall be at least 100 Mega Ohms ( $1 \times 10^8$  Ohms). In addition, there shall be no evidence of corrosion or growth of dendrites spanning 25% of the line spacing between traces. Reason for elimination (moisture condensation or debris) of more than 2 combs would require repeating the experiment.



Data analysis and modeling found that results varied mostly due to solder paste chemistry. At lower temperature (230°C) and lower oxygen < 1000 ppm), higher resistivity is seen with solder pastes “A” and “S” for 168 hours, with a similar trend at 96 hours. There is a decrease in resistivity at 5,000 and 210,000 ppm levels at 230 deg. C. As O2 ppm levels increase and low temperatures, there is a general trend towards higher flux residue on the boards that equates to lower resistivity. Figure 7 shows the effect of O2 level on SIR. This plot is only showing data where temperature is 230°C.

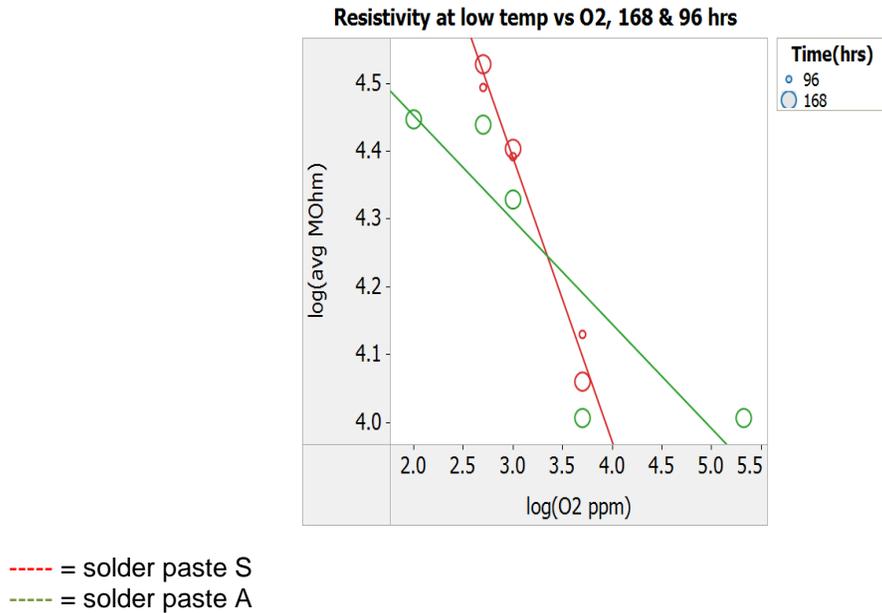


Figure 7. Resistivity plot at 230°C

### Phase II and III Programs – Wetting Test Analysis

To provide more precise measurements of wetting of lead-free solder to Organic Solderability Preservative (OSP) coated copper surfaces across varying oxygen levels and solder reflow peak temperatures the following tests were run.

A solder pattern designed to indicate the amount of wetting that occurred on a particular assembly was added to the solder paste stencil for the PCB utilized for the previous study. This pattern comprised two rows of 22 printed solder deposits. Each individual deposit is 0.64mm x 1.27mm (0.025” x 0.050”). The deposits are paired in sets of two with decreasing gaps among each pair. The gap between each pair is constant. Figure 4 shows the dimensions, in mm, of the printed deposits.

Two factors were varied during assembly of the test vehicles: Solder reflow peak temperature was set to a low peak (230°C) or a high peak (255°C), and oxygen levels were set to 100 ppm, 1000 ppm, or non-inerting (environmental). The design of experiment summarized in Table 2 shows the conditions each sample board was exposed to during solder reflow. Solder paste “S” was used for Phase II study. Two boards were assembled for each condition, and each wetting pattern consisted of two measurement sites, resulting in a total of 24 measurement opportunities. Each measurement opportunity was subjected to two separate evaluations.

The first evaluation is a count of the number of wetting pattern pairs that shorted together during reflow. As each pair of deposits is spaced further apart than the last, the number of pairs that bridge can be used to compare solder wetting under different conditions – an increasing number of bridged patterns indicates increasing wetting. Figure 8 below shows an example of a wetting pattern after reflow, with shorted patterns in the top right corner.



Figure 8. Wetting pattern, post reflow

The second evaluation is a measurement of the gap between the paste deposit pair that is spaced farthest apart. This gap will decrease during reflow as the paste wets to the underlying copper and thus a smaller gap is an indication of greater solder wetting. The measurements were performed using an Olympus SX12 stereo microscope at 40X magnification in conjunction with a SPOT Insight QE Model 4.2 camera and SPOT Advanced 3.5.5 software. Figure 9 shows an example of a paste deposit gap measurement.



Figure 9. Gap measurement

**Table 4 - Summarizes the gap measurements.**

<u>Peak Temp</u>	<u>O2 ppm</u>	<u>Board No</u>	<u>Wetting Sample Row</u>	<u>Wetting Areas Shorted</u>	<u>Gap Measurement</u>
230	100	1	Top	2	0.49
230	100	1	Bottom	3	0.49
230	100	2	Top	3	0.48
230	100	2	Bottom	2	0.45
230	1,000	1	Top	2	0.51
230	1,000	1	Bottom	2	0.52
230	1,000	2	Top	2	0.50
230	1,000	2	Bottom	2	0.48
230	210,000	1	Top	3	0.48
230	210,000	1	Bottom	3	0.51
230	210,000	2	Top	3	0.53
230	210,000	2	Bottom	2	0.49
255	100	1	Top	2	0.45
255	100	1	Bottom	3	0.43
255	100	2	Top	4	0.43
255	100	2	Bottom	4	0.45
255	1,000	1	Top	4	0.36
255	1,000	1	Bottom	4	0.35
255	1,000	2	Top	4	0.38
255	1,000	2	Bottom	4	0.33
255	210,000	1	Top	3	0.46
255	210,000	1	Bottom	4	0.40
255	210,000	2	Top	3	0.52
255	210,000	2	Bottom	2	0.51

A simple comparison of the means of the gap measurement provides some indications of the relative performance when varying one condition.

A comparison of the mean of the measurement gap for the 230° C temperature condition (**0.494 mm**) indicates that it is less effective at stimulating solder wetting than the 255° C temperature (**0.426 mm**).

A comparison of the means of the measurement gap for the 1,000 ppm atmospheric condition (**0.429mm**) to the 100 ppm condition (**0.459mm**) and the 210,000 ppm condition (**0.493mm**) indicates that the 1,000 ppm condition is the best condition to promote solder wetting.

### **Phase III Programs- Wetting Test Analysis**

Phase III study was run to provide more measurements of wetting of lead-free solder to Organic Solderability Preservative (OSP) coated copper surfaces across varying oxygen levels and solder reflow peak temperatures. Specifically, Phase III is intended to assess wetting over more reflow atmospheric and temperature conditions than Phase II using two different solder materials.

The same test board used in Phase II study was used in Phase III.

Three factors were varied during assembly of the test vehicles: Solder reflow peak temperature was set to a low peak (230°C), a medium peak (245°C), or a high peak (260°C); oxygen levels were set to 100 ppm, 1,000 ppm, 2,000 ppm, 5,000 ppm, or non-inerting (atmospheric – 210,000 ppm); “A” and “S” solder pastes were compared. In this case, a full factorial design was used, resulting in 15 experiments for each paste. Table 3 shows the conditions each sample board was exposed to during solder reflow.

As in Phase II study, the number of wetting pattern pairs that shorted together during reflow, and the gap between the paste deposit pair that is spaced furthest apart were measured. Table 5 summarizes the gap measurements.

Solder	Temperature	O <sub>2</sub> ppm	Wetting 1	Wetting 2	Gap Measurement 1	Gap Measurement 2
A	230	100	3	4	0.44	0.46
A	230	1000	3	3	0.54	0.58
A	230	2,000	3	3	0.49	0.53
A	230	5,000	3	3	0.67	0.64
A	230	AIR	1	2	0.69	N/A
A	245	100	4	5	0.51	0.42
A	245	1,000	4	4	0.44	0.54
A	245	2,000	4	4	0.56	0.54
A	245	5,000	2	2	0.59	0.55
A	245	210,000	3	4	0.51	0.67
A	260	100	4	5	0.38	0.38
A	260	1,000	3	4	0.44	0.51
A	260	2,000	4	3	0.44	0.46
A	260	5,000	2	2	0.56	0.58
A	260	210,000	4	4	0.5	0.54
S	230	100	9	6	0.3	0.34
S	230	1,000	5	5	0.42	0.38
S	230	2,000	4	5	0.47	0.42
S	230	5,000	5	4	0.5	0.54
S	230	210,000	2	1	0.54	0.58
S	245	100	9	7	0.24	0.32

S	245	1,000	6	6	0.32	0.35
S	245	2,000	5	5	0.47	0.42
S	245	5,000	4	5	0.34	0.3
S	245	210,000	2	3	0.47	0.5
S	260	100	9	7	0.2	0.32
S	260	1,000	7	6	0.26	0.26
S	260	2,000	5	4	0.49	0.37
S	260	5,000	4	4	0.51	0.47
S	260	210,000	5	4	0.56	0.58

A comparison of the mean of the measurement gap for the 230°C temperature condition (**0.502 mm**) indicates that it is less effective at stimulating solder wetting than the 245°C temperature (**0.453 mm**) and the 260°C temperature (**0.441 mm**).

A similar result occurs when comparing the means of the gap measurement assessments across peak temperatures. The 230°C temperature's average shorted gaps was **3.7 mm**, indicating that it is more effective at stimulating solder wetting when compared to 245°C temperature (**4.4 mm**) and the temperature 260°C (**4.5 mm**).

A comparison of the means of the measurement gaps across atmospheric conditions shows a proportional relationship – as the level of oxygen in the reflow atmosphere increased the gap measurement indicated decreased wetting as shown by a progressively increasing gap: {**0.359 mm, 0.420 mm, 0.472 mm, 0.521 mm, 0.558 mm**} for conditions {100 ppm, 1,000 ppm, 2,000 ppm, 5,000 ppm, 210,000 ppm} respectively.

A similar result occurs (decreasing wetting with increasing oxygen content) when analyzing the means of the shorted gap counts: {**6.0, 4.7, 4.1, 3.3, 2.9**}.

Figures 10, 11, and 12 are examples of wetting for Phase III:



Figure 10. Excellent Wetting, < 1,000 O2 ppm levels

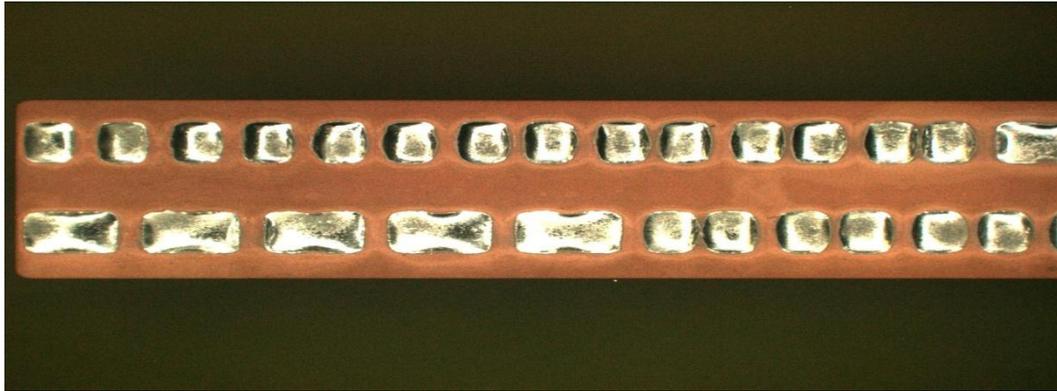


Figure 11. Medium Wetting, > 1,000 O<sub>2</sub> ppm levels and less than 5,000 ppm levels

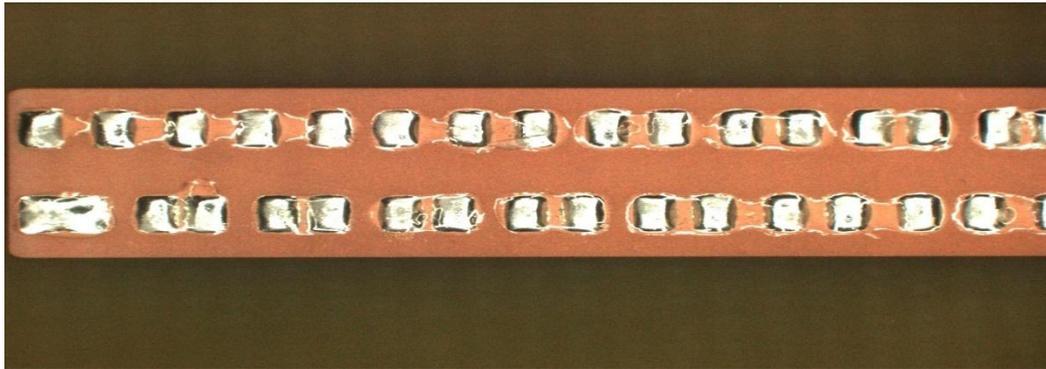


Figure 12. Poor wetting, with flux residue, high O<sub>2</sub> ppm levels > 5,000

Lower oxygen levels led to better wetting for both “S” and “A” solder paste at all three temperatures tested. Gap measured increases as oxygen is increased, indicating poorer wetting at higher levels of oxygen. On average, higher temperatures led to smaller gaps (better wetting), but the effect was not as strong as the oxygen effect. The trends were the same for both “A” and “S” solder paste chemistries, but better wetting occurred for “S” solder paste. Figure 13 shows this trend.

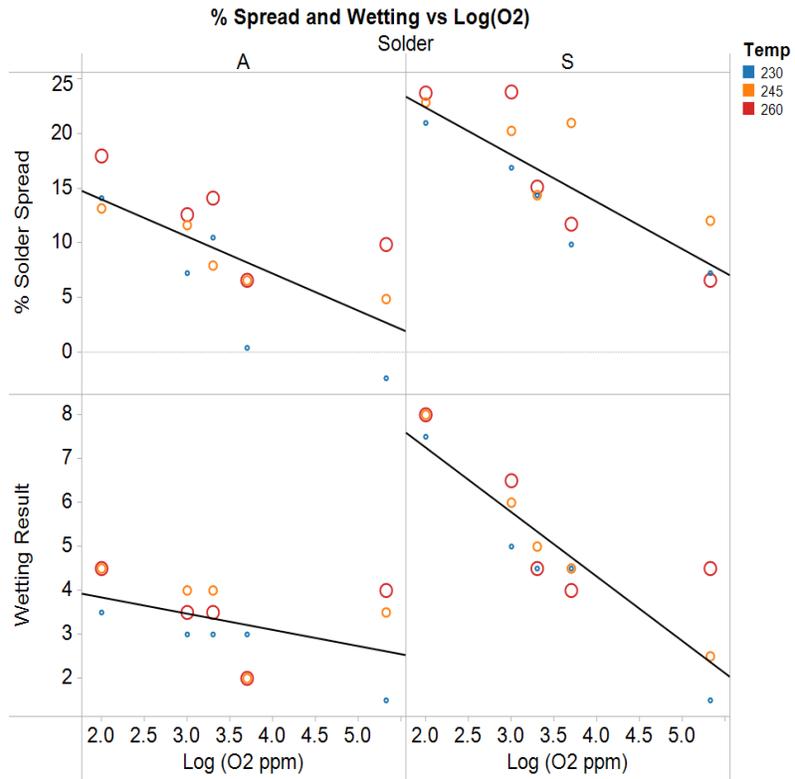
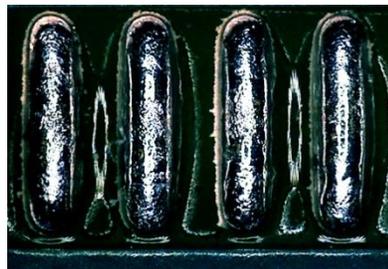
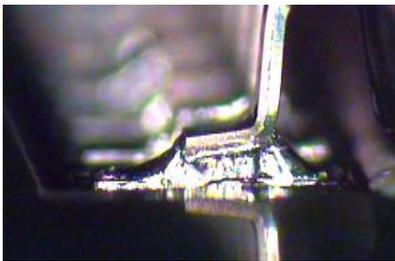


Figure 13 – Spread versus wetting

**Production Study**

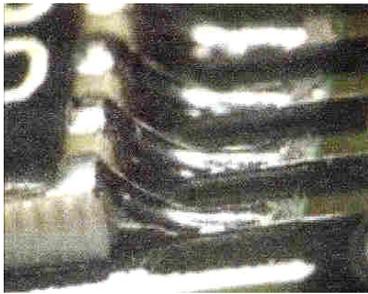
Recent production trials with our customers have demonstrated similar results that the above study has demonstrated. These figures were shared by our customers for this paper.



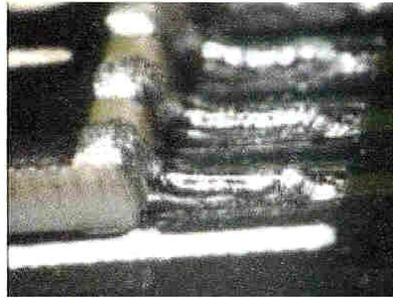
**Air processing, poor wetting and flux residue**



**N<sub>2</sub> processing, excellent wetting and no flux residue**



**Nitrogen**



**Air**

Figure 14 – Wetting – Air versus nitrogen atmosphere

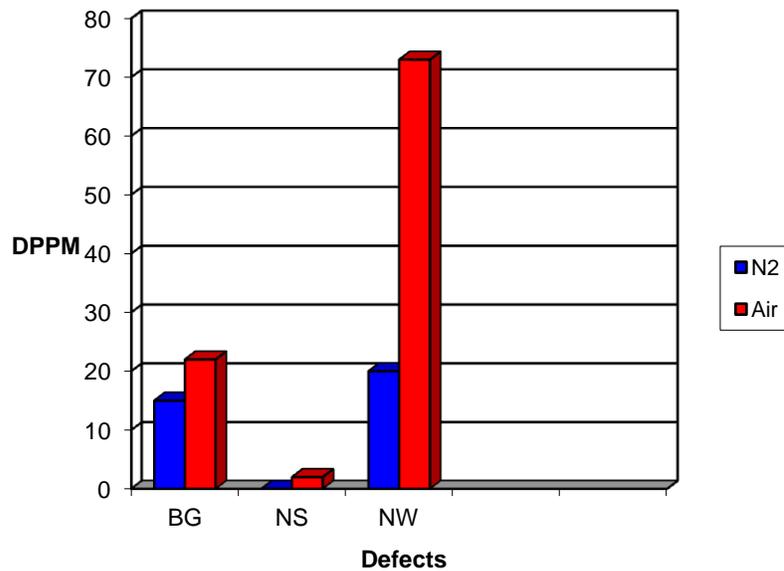


Figure 15 – Defects – Air versus nitrogen atmospheres

### **Summary**

The three studies described here demonstrate the following conclusions:

1. Nitrogen inert atmospheres have a positive effect in improving solder wetting, reduced flux residue and ionic contamination.
2. The data indicates that oxygen ppm levels of 1000 ppm or lower will provide positive solder wetting, lower flux residue and lower ionics.
3. Lower processing temperatures can be used to minimize damage to the materials of construction.

Further research is planned to better understand the effects of inert atmospheres for the ionic issues and on lowering process temperatures.

This study was contracted by Air Products and Chemicals, Inc. and conducted at the American Competitive Institute (ACI) in Philadelphia, PA. with the assistance of Jason Fullerton and Sam Pepe



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