Influence of Flux and Powder Morphology on Void Formation in Silicon Wafer Bumping

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Use of solder paste as a material to bump silicon wafers for interconnection to other level of package interconnection is a simple and cost effective process. However, the material properties of the paste become critical if the quality of the bumps is to be consistent. Especially critical for high bump quality are low solder balling and low void formation during paste reflow. Work at Kester Northrop Grumman has shown that flux material properties and powder morphology influence both the formation of voids and solder balls. This paper will describe a series of experiments that allowed an understanding of these two major problems. Follow up experiments resulted in a paste that was optimized based on flux composition and powder morphology.

Introduction

Void formation in solder pastes has been a topic of intense discussion in the electronics industry for some time. Void and solder ball formation is especially important for the bumping of wafers. Each can impact the uniformity of the bumps. Solder balls can be visually counted. Voids are detectable by X-ray. However, the ability to find the problem is not as important as the ability to control or eliminate the problem. Work at Kester has shown that the solder paste flux formulation and the powder properties have a dramatic effect on the formation of voids and solder balls. This paper reports on our work in this area and proposes some possible root causes for the formation of voids in small solder spheres.

Initial Testing

This project was initiated as a result of customer interest in a cost effective bumping procedure for silicon wafers. Although the customers had processes in place to solder bump wafers, there was a general level of dissatisfaction with these processes. A survey of customer concerns was undertaken along with a series of discussions. Based on these initial discussions with customers a 16 cell DOE, examining flux effect, atmosphere, reflow profile, and test vehicle geometry, was run to see if an obvious cause of void formation could be quickly determined. Details are shown in Table 1.

Table 1 – Initial Experimentation

Cell #	Results: Voids	Flux A	Flux B	N2	O2	Profile A	Profile B	Large Geometry	Small Geometry
1	3%	+		+		+		+	
2	3%		+		+		+		+
3	15%	+			+		+		+
4	68%		+	+			+		+
5	11%		+		+	+			+
6	22%		+		+		+	+	
7	20%	+		+			+		+
8	26%		+	+		+			+
9	31%		+		+	+		+	
10	7%	+			+	+			+
11	10%		+	+			+	+	
12	1%	+			+		+	+	
13	3%	+		+		+			+
14	4%		+	+		+		+	
15	3%	+			+	+		+	
16	5%	+		+			+	+	

Testing was done using fine (small geometry) and coarse (large geometry) pitch silicon test wafers. Paste printing was done by hand. Voids are expressed as the percent of all bumps examined that contained voids. (Voids were determined using a FeinFocus X-ray system Fox-106.25). A statistical analysis of the data, using the SAS Institute "JMP" statistical analysis software package, concluded that none of the factors could be judged statistically significant. All pastes were made with 63/37 Sn/Pb Type 6 solder powder.

Given the lack of conclusive results in the first experiment a second DOE was run to determine if the laboratory wafer bumping process and x-ray test method used could provide a reasonable level of statistical confidence as regards the detection of voids and their relation to formulation used. The number of variables was reduced and only paste type and the geometry of the test wafer were considered.

The details of the second experiment are shown in Figure 1.

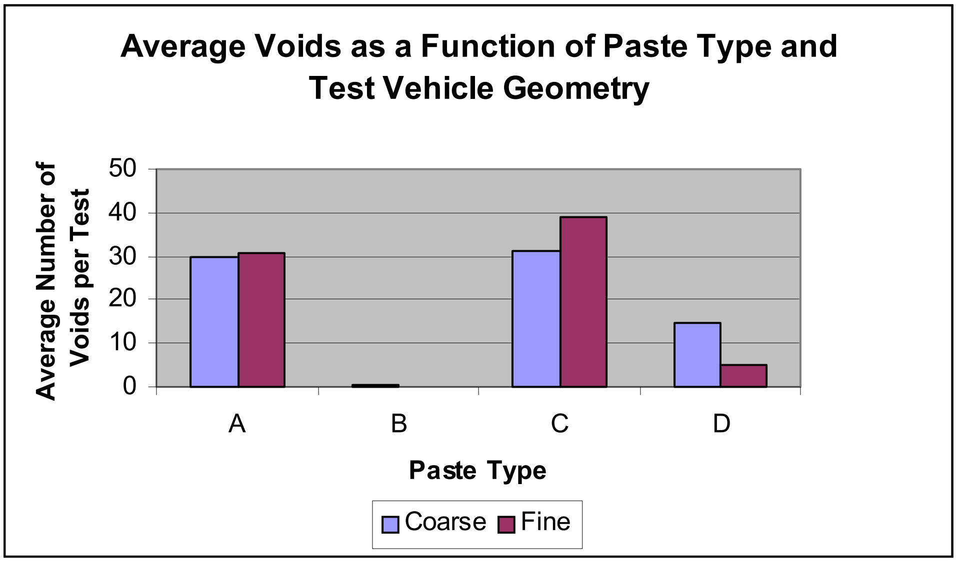

Figure 1 – Effect of Paste Type and Test Vehicle Geometry

Statistical analysis of the data using "JMP" showed that there was a significant difference between paste types, but none between fine and coarse geometry test vehicles for a given paste type. This shows that this void test method is statistically valid and could be used in further paste development.

Test Discussion

Data provided by customers seemed to indicate that the solvent system used in the formulation was the primary contributor to solder ball formation. An attempt was made to expand this line of thinking to the formation of voids. However, examination of the formulations above did not provide a quick sense of why B and D were lowest in voiding. A review of information available on the Internet on coalescence of bubbles in liquids provided an interesting link to a thesis from the Norwegian University of Science and Technology (L.Hagesaether, "Coalescence and Breakup of Drops and Bubbles", March 2002)

Two items of interest were gleaned from the thesis:

- Formation of a rigid surface at the top of a liquid drop will impede the break up of a bubble that has risen to the surface.
- A coating on the surface can speed or impeded the break up of a bubble that has risen to the surface.

These two items may be interpreted, at first glance, as affecting bubble (potential void) breakup through:

- Oxide formation on the surface of a reflowed solder paste dot and
- Flux solvent/surfactant effects on the molten solder's surface during reflow.

This would imply that the properties of the flux/molten solder surface interaction might give a clue as to the propensity of a system to form voids. Void formation (or non-formation) should be a direct result of the surface thermodynamics (energetics) of the flux/solder system. In this case the surface thermodynamics, which influence whether the surface of the molten metal is wetted well or poorly by the flux system, should provide a visual means of assessing the quality of the flux/solder interaction.

Examination of Flux Wetting

To test whether the thermodynamic model could be correct a series of visual evaluations of the 4 solder pastes that had been tested by x-ray for voids was conducted. Visually, in as far as a melting of a paste sample on a copper coupon could tell, the fluxes that best wet the molten solder surface were B and D. This matches the results of the x-ray analysis although this wetting test is not a statistically valid test.

This test was refined somewhat by printing (using the stencil used for testing paste spread) three dots on a copper coupon and reflowing the coupon in air and nitrogen. Visually looking at three coupons each of the fluxes A, B, C and D confirmed that paste D did remain over the solder surface while C visibly de-wets from the surface. Interestingly though, C did appear to also wet the solder surface and leave a residue (that could be confirmed by touching the surface) on the solder.

Accordingly an X-ray analysis of the four paste prints was done under two atmosphere conditions, air and nitrogen. As before the pastes were made with 63/37 Sn/Pb Type 6 powder. The prints were not on wafer test vehicles. Instead the printing was done on copper coupons using stencil for IPC Solder Ball Test Method 2.4.43. The thought here was the wetting of the surface should be influenced by the atmosphere and may differ significantly for the various pastes thus affecting the tendency of a paste to void. Figure 2 is a summary of a manual count of the voids in one of the test prints under the conditions of air and nitrogen respectively for pastes A, B, C, and D.

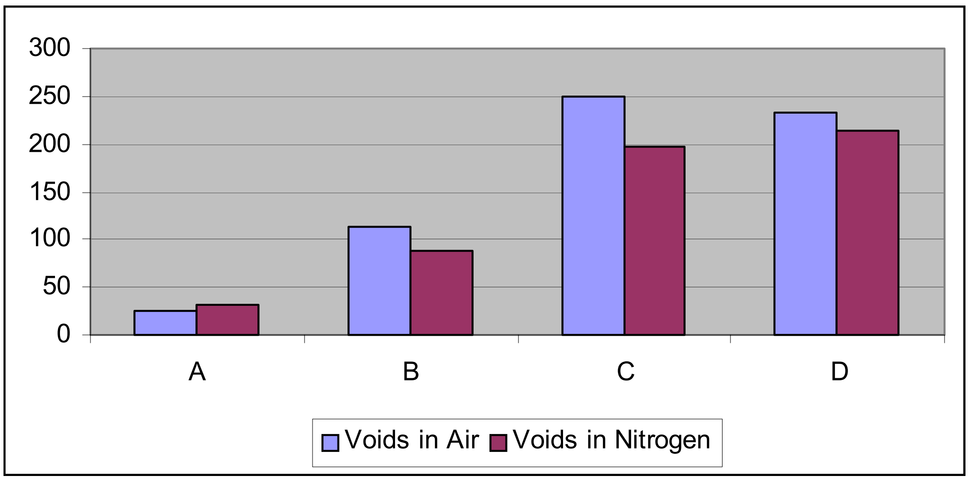

Figure 2 – Effect of Atmosphere on Void Formation

Given the small number of repetitions, these measurements are not statistically significant enough to use for the drawing of any conclusions. However, it is interesting to note that at this larger geometry paste D is no longer one of the better performers. It is also interesting to note that A, which has been confirmed by customers as being low voiding in BGA assembly, did well in this voiding test and poorly in the test with the wafer bumping test vehicles (above). As the geometries of the two test vehicles are very different it is quite probable that an effect associated with the radius of curvature of the solder melt is at work here.

Statistical Reproducibility of Large Print Void Testing: Link to Activators

The test results of the table above represent only one replication per cell. Given that we have confidence in the ability of the wafer level reflow and x-ray analysis to correlate void formation to formulation, and we wish to use the simpler and cheaper print test on copper coated FR-4 coupons, it is important that we have the same level of statistical confidence. Accordingly a four-cell test of activators was again run with two controls using the IPC Test Method 2.4.43. Each cell had 9 replications. (Some cells ended up with only 8 measurements because of poor print on some paste dots. These dots were ignored.) Two controls were also run to insure that there is a valid link to previous work.

Manual counting of voids produced was done along with a follow up analysis of the manual count data using "JMP" statistical program. The statistical analysis did show that, given the number of repetitions per cell (nine), the manual method was able to statistically distinguish the solder pastes on the basis of their voiding behavior. However, the relative ordering of the voiding was the same as shown in Figure 2, i.e. the geometry of the print appears to impact the tendency of a paste to void.

Interim Conclusions

The following conclusions can be drawn from the tests described above:

- The X-ray testing for voids using the available wafer test vehicles is statistically valid.
- Use of either the coarse or the fine feature vehicle is acceptable: both sets of results are statistically equivalent

- Void formation is formulation dependent.
- The attempt to develop a simpler test using IPC TM 2.4.43 and a copper coated FR-4 test vehicle has given some indications that such a modified test may be possible. However, the results do not agree with those obtained with the wafer bumping test vehicle.
- Void formation is geometry dependent. This appears to be due to the larger solder surface area relative to the wafer test vehicle.

Other tentative, but not statistically validated, conclusions are:
- Void formation is related to the degree of wetting of the solder melt by the flux vehicle.
- Nitrogen reflow reduces void formation.

It is worthwhile to note that the velocity of rise of a bubble in a continuous phase, as described by the Stokes relationship, (E.S.R.Gopal, "Principles of Emulsion Formation" in Emulsion Science**,** P.Sherman**,** Ed., Academic Press, 1968) is directly proportional to the square of the radius of the bubble, i.e. the smaller the bubble the lower the rate of rise. Thus for smaller geometries small bubbles will break slower than for a larger "pool" of solder.

In addition we should consider that the two behaviors we are interested in, void formation and solder ball formation, might be two manifestations of the same phenomena. Consider that solder balls represent dispersed solder whereas voids represent dispersed flux. Such a phenomena may be related to the familiar oil-in-water/water-in-oil inversion points demonstrated by some oil/water/surfactant systems.

Formulation Tests

The above discussions of what may influence void formation and what methods are acceptable for measuring void formation are interesting and suggestive as to lines of new investigation but insufficient for answering a customer's specific concerns about a particular material/formulation. Based on previous work an 18 cell DOE looking at the influences of activator, solvent and vehicle was run. All pastes were made using 63/37 Sn/Pb Type 6 powder.

Testing was done using coarse pitch silicon test wafers. Paste printing was done by hand. Voids are expressed as a percent of bumps with voids based on number of bumps examined. Solder balls were also determined via x-ray with a confirmation done using an optical microscope. Solder paste quality was a subjective assessment of the printability of the paste done on a 0 to 5 scale with 5 being preferred. The results of the void counting and solder ball formation were examined using the "JMP" test program. Data from the testing is shown in Table 2.

Table 2 – Experimental Design Examining Flux Composition

Flux	Average Voids per Bump	Vehicle	Solvent Sys	Act Sys	Solder Balls?	Paste Quality
1	0.87	B	A	A	No	3.5
2	0.5	A	B	B	Yes	5
3	0.8	A	A	A	No	2
4	0.58	B	B	A	No	3.5
5	0.25	B	A	B	Yes	5
6	0.57	A	B	A	No	3.5
7	0.28	A	A	B	Yes	4
8	0.56	B	B	B	Yes	5
9	0.13	C	B	B	Yes	0
10	0.03	C	B	A	No	0
11	0.16	C	A	B	Yes	0
12	0.35	C	A	A	No	0
13	0.83	B	C	A	No	3.5
14	0.67	B	C	B	Yes	5
15	0.78	A	C	A	No	3.5
16	0.02	A	C	B	Yes	5
17	0.09	C	C	A	Yes	0
18	0.15	C	C	B	No	0

"JMP" analysis of the factors varied in the testing is summarized in Table 3.

Table 3 – Flux Composition Effect on Paste Properties

Factor	Significant for Voids?	Significant for Solder Balls?	Significant for Viscosity?
Vehicle	Yes	No	Yes
Activator	Yes	Yes	Yes
Solvent	No	Slight (w/ Activator)	No

The analysis indicates that the most significant factor for void formation and subjective print quality is the vehicle followed by the activator. The solvent is the least influential material. Solder ball formation is activator dependent with a small interaction between the activator and solvent that may be useful.

Response Surface

Based on the above work it was decided to focus on the effect of vehicle. A DOE based on a three material response surface was constructed. The outline and results of that test are shown in Table 4. (The solvent and activator systems chosen for the experimental pastes were standard. Two standard production pastes, labeled A and B in the table, were also run as controls.)

Table 4 – Vehicle Response Surface Trial

Paste	Vehicle C	Vehicle A	Vehicle B
A	NA	NA	NA
B	NA	NA	NA
19	80%	20%	
20		20%	80%
21	60%		40%
22	40%		60%
23	20%	80%	
24		80%	20%
25	20%	60%	20%
26	40%	40%	20%
27	20%	40%	40%
28	60%	20%	20%
29	20%	20%	60%

Results of the analysis for voids in the test vehicles prepared with these pastes are shown in Figure 3.

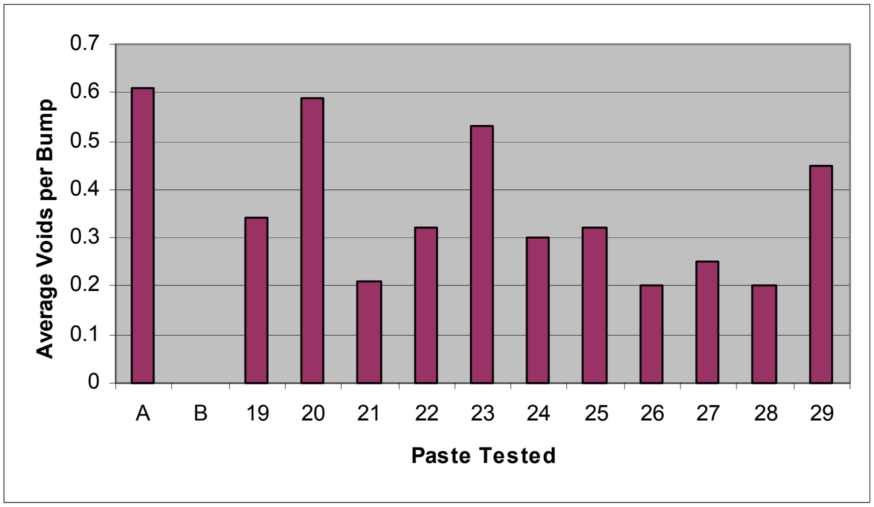

Figure 3 – Flux Response Surface and Voids Found

The purpose of this test was to see the effect of activator. As can be seen in the figure above, variations in the vehicle had a dramatic influence on the number of voids formed. From this response surface further modification of pastes 21, 24, and 25 were proposed. The modifications centered on use of two enhanced activator systems labeled herein as HA and AFA. The samples were prepared and tested for solder balls and voids. The results for pastes in the Figure 4 show that activator system HA improved void performance.

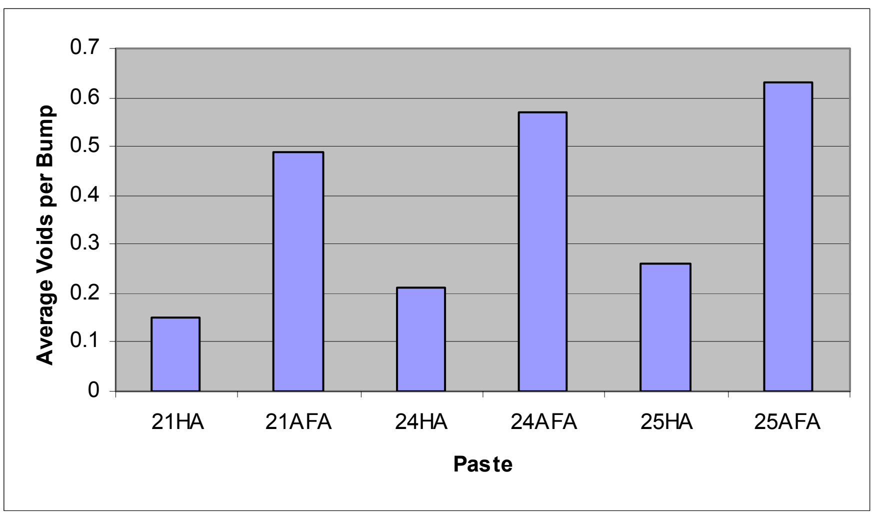

Figure 4 – Effect of Activator on Voids

Accordingly it was agreed that samples of the standard product would be provided to the customer for their evaluation. Results from the customer's trials agreed with the laboratory evaluations. This suggested that the focus for further development should remain fixed on the vehicle and activator packages of paste 21HA.

The above test was then repeated using the best performers, 21HA, and paste 11 from the previous experiment. The three "ideal vehicle" samples (based on the response surface experiment described previously) were also prepared. A description of the vehicle modification experiment and results of the test for voids are shown in Table 5 and Figure 5. The "best" vehicles were tested along with 3 production pastes with modified vehicle packages.

Table 5 – Vehicle Composition

Paste	% Vehicle C	% Vehicle A	% Vehicle B
21HA	60%		40%
11HA	40%	40%	20%
Paste A	3%	74%	23%
Paste B	74%	13%	13%
Paste C	74%		26%

The best candidates from the previous experiments were then tested on coarse and fine pitch geometry micron wafers using a standard product flux as a control.

Results are shown in Figure 5.

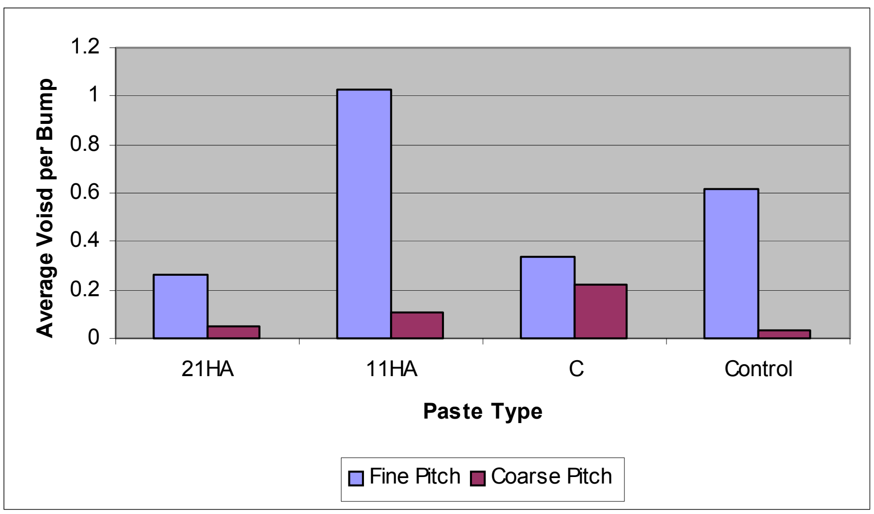

Figure 5 – Voids as a Function of Vehicle Composition

Again 21HA performed best of the samples tested. However, statistical analysis of the results indicated that pitch geometry has a dramatic effect on void formation for all pastes tested. Specifically the finer pitch is much more prone to voiding than the coarse pitch test vehicle. This result is in agreement with results of the initial paste development work.

To determine the contribution of processing or fabrication an experiment was performed. Two key processing variables were examined: production lot-to-lot variation and reflow profile condition. Results are shown in Figure 6

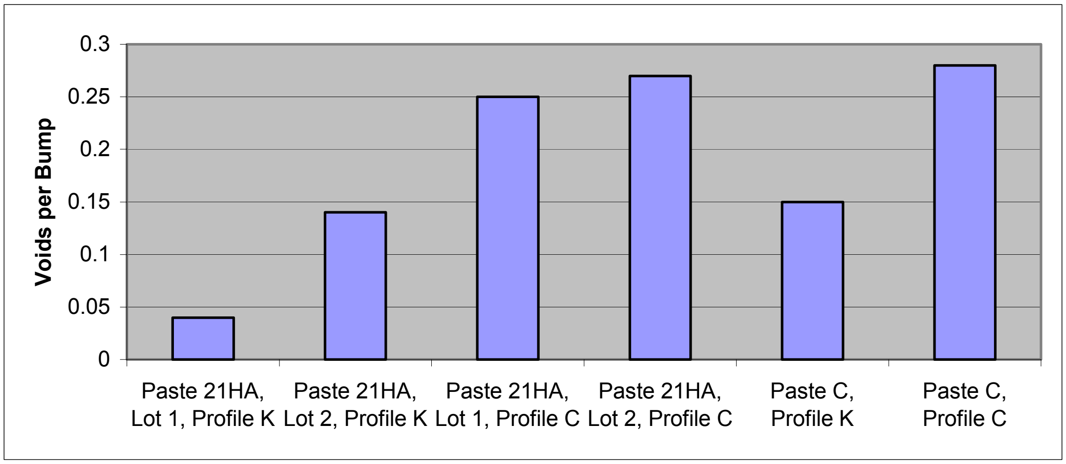

Figure 6 – Effect of Lot and Reflow Profile on Voids

The results obtained show that paste 21HA is low voiding compared to previous materials tested. In addition, neither the reflow profile nor the lot tested showed statistically significant effects for the number of repetitions run.

Powder Type Investigation

At this stage the effects of vehicle, activator, and test vehicle geometry were statistically confirmed. However, feedback from samples tested by customers suggested that powder and alloy type also have a strong influence on void formation. To test these nine 63/37 type 6 powders from various vendors, with various powder manufacturing techniques were prepared as pastes using both 21HA and a typical water-soluble paste flux that we will call WS#1. The controls were Sn63 type 3 powder with no clean 21HA and the water-soluble flux, WS#1. A total of 20 samples were made using the above formulas. The paste quality was judged based on texture and smoothness. All pastes were printed on the fine geometry wafer test vehicle and reflowed using the standard reflow profile and a Nitrogen atmosphere. The FeinFocus X-ray examination was used to detect voids.

Not all of the 20 samples reflowed. The water-soluble samples, except for the sample made using Type 3 powder and one of the Type 6 powders, did not coalesce to form bumps. This may be a result of insufficient activation for the larger surface area of the type 6 powders. Figure 7 shows the void count as a function of powder vendor for the two fluxes, 21HA and WS#1, tested.

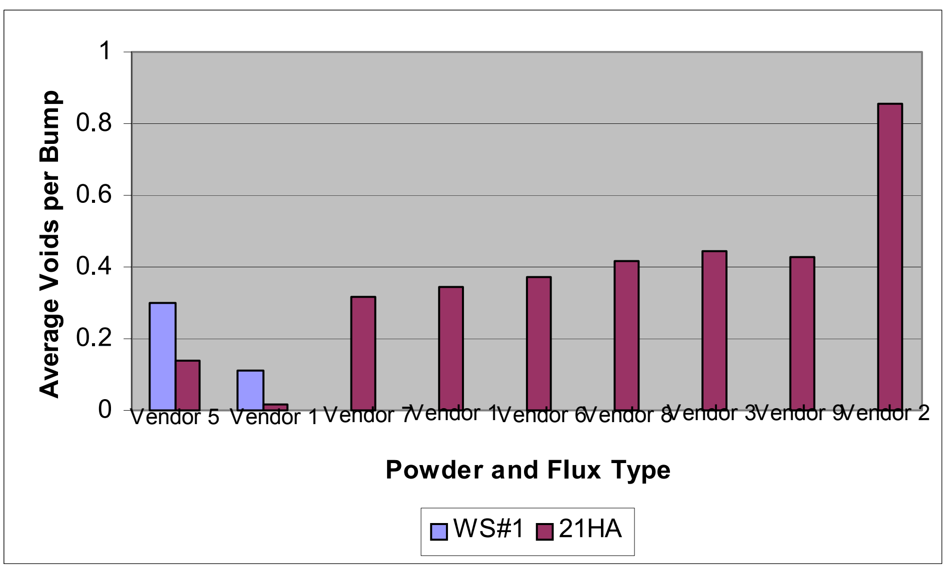

Figure 7 – Voids by Powder Lot for Two Fluxes Tested

Note that there is a large difference in the void performance of the various powder types. This performance is strongly flux dependent, e.g. Vendor 5 Type 6 performs well with WS #1 and very poorly with 21 HA.

This experiment was then repeated with a second water-soluble flux paste, WS#2, and the powder types used in the experiment above. Results are shown in the Figure 8 for four vendors and a Type 3 powder control. (All powders are 63/37 alloys.)

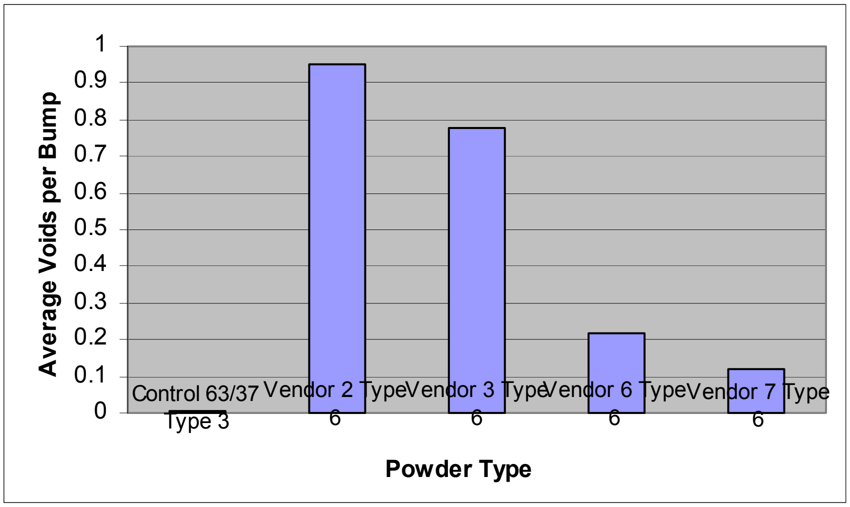

Figure 8 – WS#2 and Voids as a Function of Powder Vendor

Some comments on the results are in order:

- Paste flux type is impacted by powder type. Each paste flux, 21HA, WS#1, and WS#2, have a "preferred" powder that gives lowest void formation.
- Correlation between paste quality (as assessed qualitatively by smoothness of the samples after mixing) and voids was inconclusive, e.g. samples with good paste quality had high voids and vice versa.
- The relationship between powder size distribution and voids is reproduced for all three flux pastes, i.e. larger powder size produces fewer voids.

Given that the powder type has such a great influence an attempt was made to link void formation to other measurable quantities of the paste/powder. Tests were run using both the IPC solder ball test and an in house optical test device that measures the relative reflectivity of the solder powder. This equipment is called the "Gray Scale Inspection System" and is based on a Cognex vision system. Results for this "Grey Scale Analysis" are shown in Table 6 along with void measurements.

Table 6 – Gray Scale Measurements and Voids

POWDER TYPE (by vendor number)	**IPC SOLDER BALL TEST (21HA)**	**IPC SOLDER BALL TEST (WS#1)**	**VOID RESULTS (21HA flux)**	**VOID RESULTS (WS#2 flux)**	**GRAY SCALE ANALYSIS**
Type 3 (Control)	**Preferred**	**Unacceptable**	**0**	**0.007**	**67.31**
2	**Acceptable**	**No reflow**	**0.445**	**0.951**	**43.22**
3	**Acceptable**	**Unacceptable**	**0.373**	**0.778**	**60.41**
4	**Unacceptable**	**No reflow**	**-**	**-**	**43.55**
5	**Acceptable**	**Unacceptable**	**0.858**	**-**	**64.05**
6	**Acceptable**	**No reflow**	**0.315**	**0.215**	**44.18**
7	**Acceptable**	**Unacceptable**	**0.139**	**0.122**	**65.16**
10	**Acceptable**	**Acceptable**	**0.429**	**-**	**49.56**
11	**-**	**Preferred**	**-**	**0.387**	**65.11**

Two plots of the gray scale values versus the voids for 21HA and WS#2 are shown in Figures 8 and 9. (A higher gray scale number implies that the powder is more reflective, i.e. brighter and shinier.) Although the gray scale test had proved in the past to be an accurate measure of oxidation level for both lead free powders and solder pre-forms one can note that there is no correlation between the void results and the gray scale for either flux.

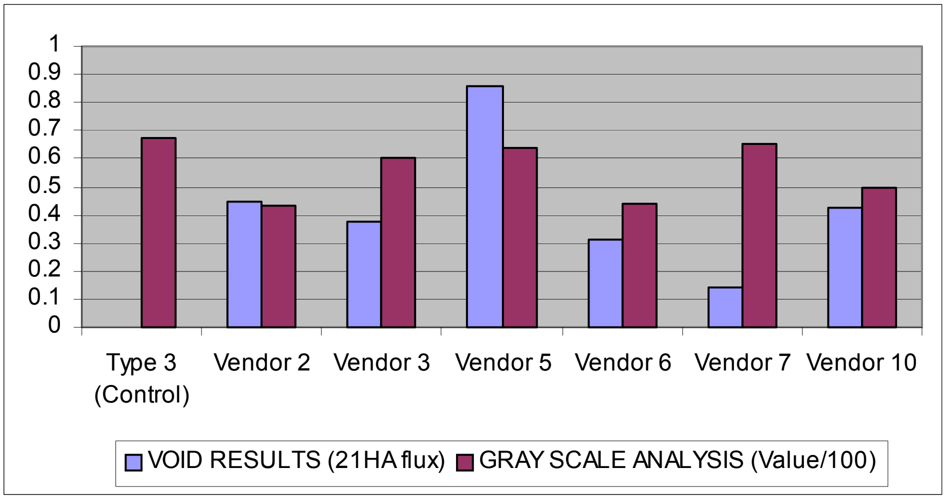

Figure 9 – Voids by Powder Vendor and Gray Scale of Powder for 21HA

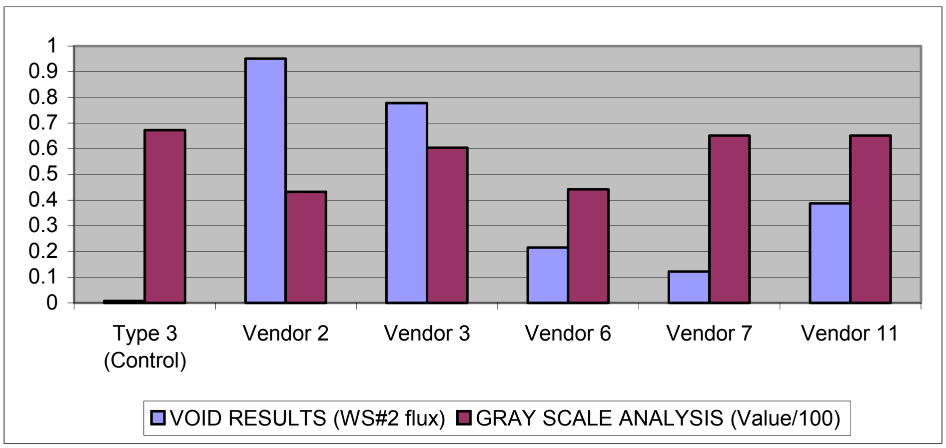

Figure 10 – Voids by Powder Vendor and Gray Scale of Powder for WS#2

Percent Flux and Effect On Voids

If powder size effects voids, i.e. smaller powders give higher void counts, this effect may simply be the result of a larger surface area for the smaller sized powders. It is possible that this effect could be circumnavigated by simply using a higher proportion of flux to powder.

An initial test of this hypothesis was done using 21HA paste flux, Vendor 5 Type 6 63/37 powder and flux percentages (by weight) of 10.5 (the standard flux percent), 12.5, 15, 21 and 31.5. Results of the JMP analysis indicate that increase above the standard 10.5 % by weight flux result in a statistically significant decrease in the level of voiding.

A separate test with WS#2 paste flux and Vendor 5 Type 6 63/37 powder was also run. As for the 21HA, WS#2 demonstrates a statistically significant void reduction as flux pastes percent by weight increases. However, this reduction in voids occurs only at a flux percentage of 29.3% and greater.

Effect of Powder Oxide Formation

If either available flux per powder surface area or less powder surface area per flux available reduces voids it is possible that void formation is a surface dependent phenomena. This hypothesis is reinforced by the dependence of void formation on powder type and specific flux interaction. Accordingly it is possible that modification of the surface may affect void formation. The easiest surface modification is oxidation.

Although the initial tests of void formation and oxide level, as measured by the gray scale analyzer, showed no correlation, a second trial was run for confirmation.

Vendor 5 Type 6 63/37 powder was placed in a 130ºC oven for periods of 1 hour, 3 hours and 24 hours. A series of pastes, using both 21HA and WS#2, were made using these powder types at 10.5% by weight paste flux. These paste were printed on the fine geometry wafers and reflowed in Nitrogen. Results are graphed in Figures 11 and 12. Notice that aging time has no effect with the 21HA but has a definite effect with the WS#2.

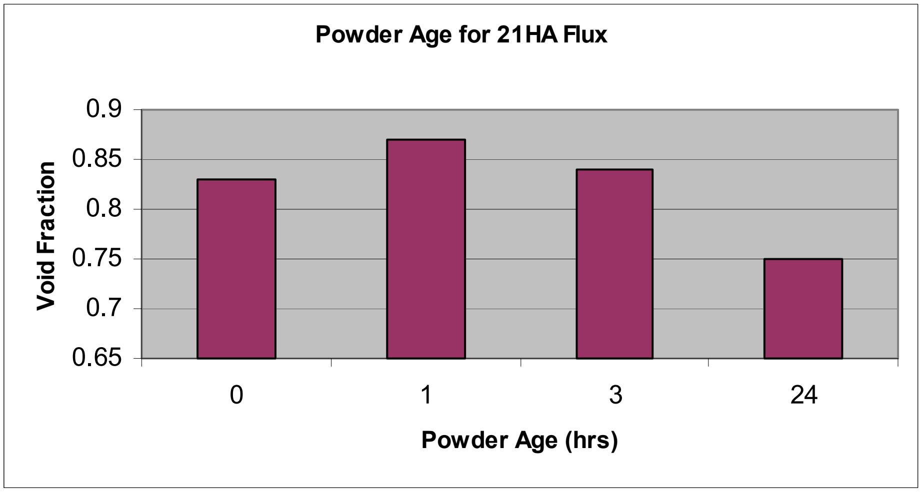

Figure 11 – Effect of Powder Age on Voids for 21HA

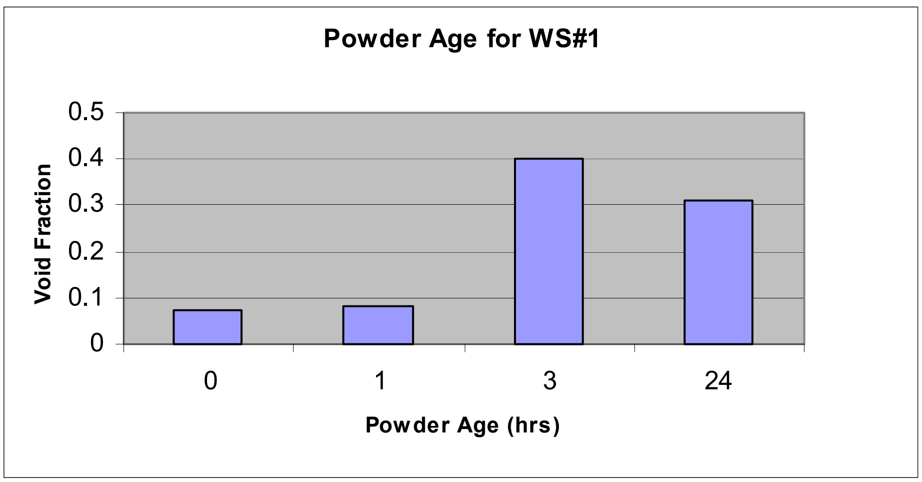

Figure 12 – Effect of Powder Age on Voids for WS#1

Statistical analysis showed that for the 21HA flux there is no effect on voids associated with powder aging. However, WS#2 begins to show increased voids at 3 hours of aging. This aging effect is magnified with further aging such that, statistically, the number of voids is significantly different for the two aging groups of zero and one hour and three hours and twenty-four hours.

Conclusions

The tests described above indicate that the formation of voids in the wafer bumping process is the result of a complex interaction of several factors. Those factors include:

- The vehicle system used in the flux,
- The activator system used in the flux,
- The geometry/size of the bump being formed,
- The total powder surface area relative to the amount of flux available in the paste,
- The source of the powder used in the paste,
- And the interaction of the specific flux with the oxides on the powder surface.

At present there is no " a priori" test that can provide a quick assessment of which powder is best matched to which flux in order to give low voids in wafer bumping. However, work is now underway at Kester Northrup Grumman to look at the thermodynamics of the wetting of the melting solder by the flux during the reflow process. The strong correlation between print size and void formation indicates that surface phenomena/interactions, not yet completely defined, may be the primary causes of solder voiding in wafer bumping.

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