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An Assessment of the Suitability of Current PCB Laminates to Withstand Lead-free Reflow Profiles

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ABSTRACT

Five different aspects of PCB manufacture and processing have been investigated to explore whether the move to the hotter reflow profiles associated with lead-free soldering would affect the performance of four common laminate types (i.e. BT epoxy; FR4; polyimide; high Tg epoxy). Boards of the different laminates were subjected to multiple cycles of temperature profiles representative of lead-free soldering, and then studied in relation to dimensional stability, bend testing, surface insulation resistance, surface composition and solderability

The work has highlighted two areas of concern if current laminate types and immersion gold pcb finishes are used in lead-free soldering technologies. First, if a board is going to be subjected to stresses during the lead-free soldering operation, then manufacturers wishing to have a flat, or near flat, assembly after soldering, should consider whether or not the Tg of the base laminate material is appropriate. Second, the lower soldering yields after more than a single lead-free soldering operation, suggests that more aggressive fluxes must be used to maintain soldering yield.

The increased temperatures of lead-free soldering had little or no effect on dimensional stability or surface insulation resistance of the laminate materials studied.

1. INTRODUCTION

Driven by consumer pressures and legislation, the global electronics industry has begun the move to eliminate lead from its manufacturing processes. Far eastern companies in particular are being driven largely by consumer preferences for environmentally friendly products in their home markets. They also have an eye to their European markets where impending European legislation in the form of the WEEE (Waste of Electronic and Electrical Equipment) Directive and the ROHS (Restrictions On the Use of Certain Hazardous Substances in Electronic and Electrical Equipment) Directive are leading Europe into a complete ban on the use or disposal of lead in most electronics manufacturing industries by 2007 (currently). Many observers would not be surprised to see this timescale foreshortened. Hence, many Japanese companies, such as Fujitsu, Hitachi, Matsushita, Mitusbishi, NEC, NTT, Sony and Toshiba have active lead reduction programmes, many with the aim on being lead-free by the end of 2002

The industry has already made great strides towards finding alternative alloys for solder (SnPb), and for lead-containing printed circuit board finishes, and is beginning to address the issue for component finishes. The alternatives proposed to replace SnPb solders, are generally new alloys that have not previously been widely used within the electronics industry, so little knowledge of their performance as engineering materials is available. The alternatives for printed circuit board (PCB) finishes have largely been available for some time and these include immersion gold on nickel, organic solderability preservative, and immersion silver, and end-users have considerable experience with these alternatives. However, the majority of this experience lies in processing PCBs through reflow profiles optimised for SnPb solders. With the introduction of lead-free joining materials, the reflow profile is expected to be some 30^{o}C hotter (Reference 1). Little experience is available on how the laminate material and the surface finish of the PCBs will behave when subjected to these increased temperatures.

Of particular concern is the use of current materials in high reliability or safety critical applications. Although high reliability electronic markets such as military and aerospace are not currently mentioned in WEEE or ROHS Directives, the move of the majority of the manufacturing marketplace away from lead-containing products will result in a dearth of product development with lead-containing interconnection materials. Hence many high reliability users may feel forced to change to lead-free materials to ensure continuity of supply and up-to-date materials development. They, along with manufacturers of equipment listed under these Directives, are now concerned about the reliability of PCBs after the higher temperature processing associated with lead-free processing. Until such data are widely available and understood, the introduction of many products incorporating lead-free alloys will be delayed with the corresponding economic impact on UK industry, particularly if industries in other parts of the world gain commercial advantage through earlier introduction of lead-free products.

To address these concerns the National Physical Laboratory has initiated a review of the performance of four common PCB laminate types after being processed through multiple typical SnPb and lead-free processes.

2. SUBSTRATE DESIGN AND REFLOW CONDITIONING

2.1. PCB test vehicle design

A PCB test vehicle (TB40) incorporating a number of test patterns was designed, and a photo is shown in Figure 1. The structure was designed so that each of the test coupons (A, B, C, D, E and F) could be separated from each other after processing, and evaluated individually.

Test coupons A, B and C were test pads for globule solderability testing. Coupon D had test strips for solderability dot testing. Coupon E was intended for bend testing, and coupon F contained surface insulation resistance test patterns. The crosses in the copper tracking were used for measurement of dimensional stability.

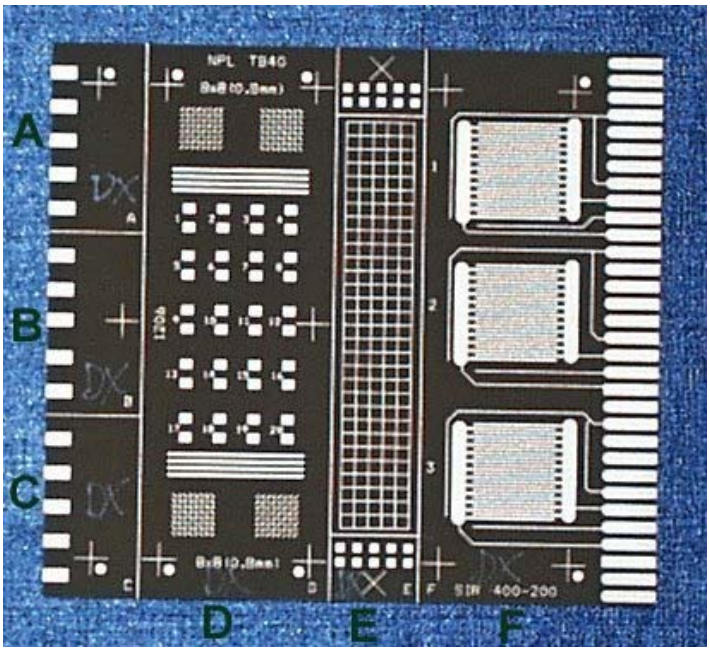

Figure 1: TB40 test board

2.2. PCB base laminate types

The test boards were all manufactured using a single facility. Also the samples were finished with immersion gold on electroless nickel. Four different laminates were chosen and these were:

FR4: A glass-fibre reinforced epoxy laminate with a glass transition temperature, Tg, of around 140^{o}C, the most commonly used substrate for surface mount assembly

High Tg Epoxy: Similar to FR4 but with a higher glass transition temperature in the range of 180^{o}C. Some users report that laminates of this type can be more susceptible to thermal degradation as compromises may have been made with the resin systems to make these materials process in a similar fashion to FR4 (Reference 2).

BT Epoxy: A glass-fibre reinforced laminate with a resin matrix, which is a blend of bismaleimide/triazine, and epoxy resin giving enhanced thermal, mechanical and electrical performance over most epoxy materials. Such systems have high glass transition temperatures in the range of 180 -200^{o}C.

Polyimide: A glass-fibre reinforced polyimide based laminate with a glass transition temperature of around 260^{o}C, giving greater thermal performance over epoxy-bimaleimide resin blend systems.

The four types of assembly are shown together in Figure 2.

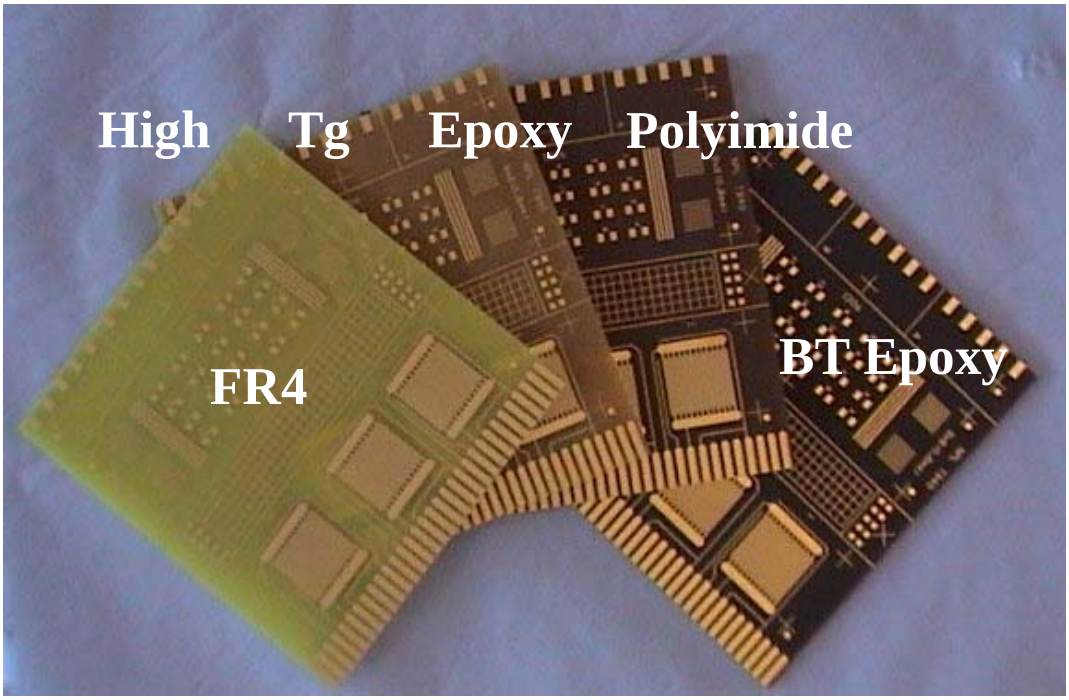

Figure 2: Photograph of four laminate types

2.3. Reflow conditions

In order to simulate the lead-free soldering profile, samples of each laminate type were subjected to a five zone convection reflow soldering system. Additionally, in order to accelerate any detrimental effects of the higher temperatures, each board was processed four times. The PCBs were allowed to cool to room temperature after each pass. The profiles were measured by attaching three thermocouples at regularly spaced intervals to a PCB and monitoring the temperatures reached as they passed through the reflow oven. Profiles were subsequently downloaded to a PC for analysis. The profiles for SnPb conditioning (Figure 3) and lead-free conditioning (Figure 4) are shown below. The main differences between the profiles are highlighted in Table 1.

Table 1: Comparison of conditioning profiles

	SnPb profile	lead-free profile
Max temp reached (degC)	219	253
Average Time above 185°C (secs)	56	140
Average Time above 217°C (secs)	5	62

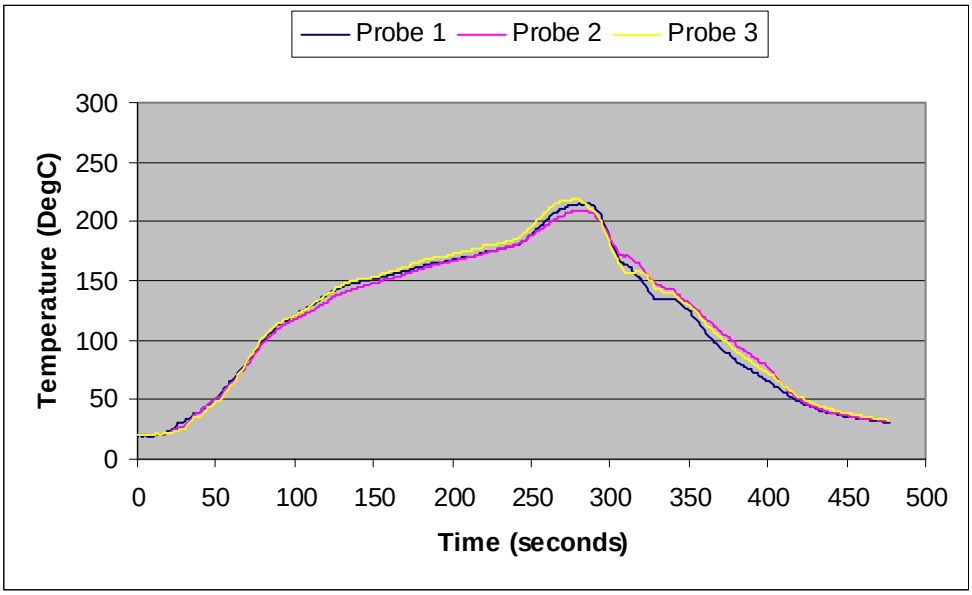

Figure 3: SnPb conditioning profile

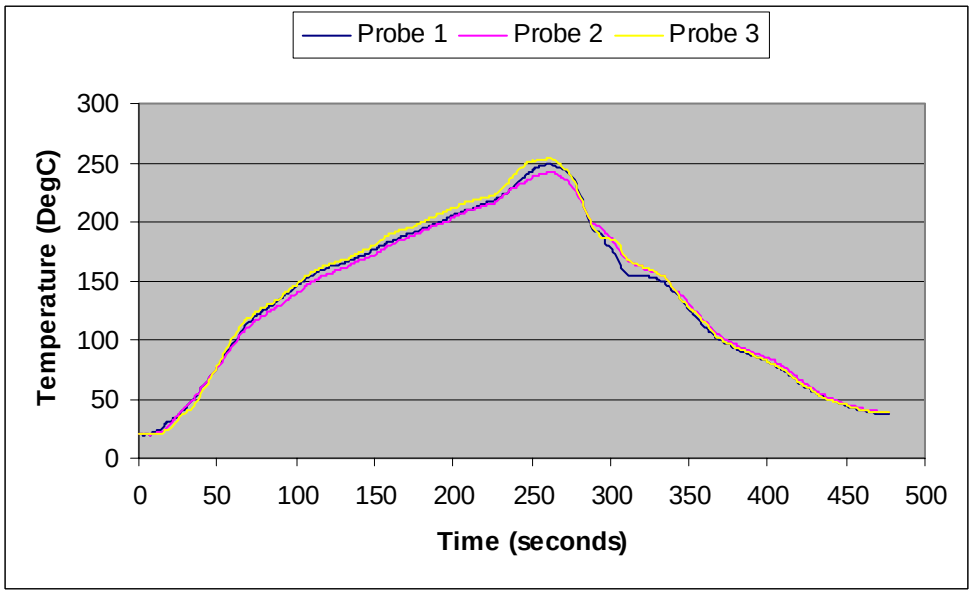

Figure 4: lead-free conditioning profile

After conditioning, the following tests were undertaken on all four types of laminate:

- Dimension stability
- Bend testing at elevated temperatures
- Surface insulation resistance testing
- Auger surface analysis
- Globule solderability testing
- Dot solderability testing

3. DIMENSIONAL STABILITY

3.1. Procedure

Prior to conditioning, dimensional measurements were carried out on PCBs of each laminate type, using a travelling microscope. The distance between the inside of the crosses in copper on the PCBs was measured as indicated in Figure 5. After conditioning through two and then four lead-free profiles (as detailed in section 2.4), the PCBs were measured again to assess if there were any dimensional changes. Each measurement was taken after the PCBs had been allowed to return to room temperature and humidity over night. As lead-free conditioning was considered to be a worse case, no SnPb conditioning was undertaken.

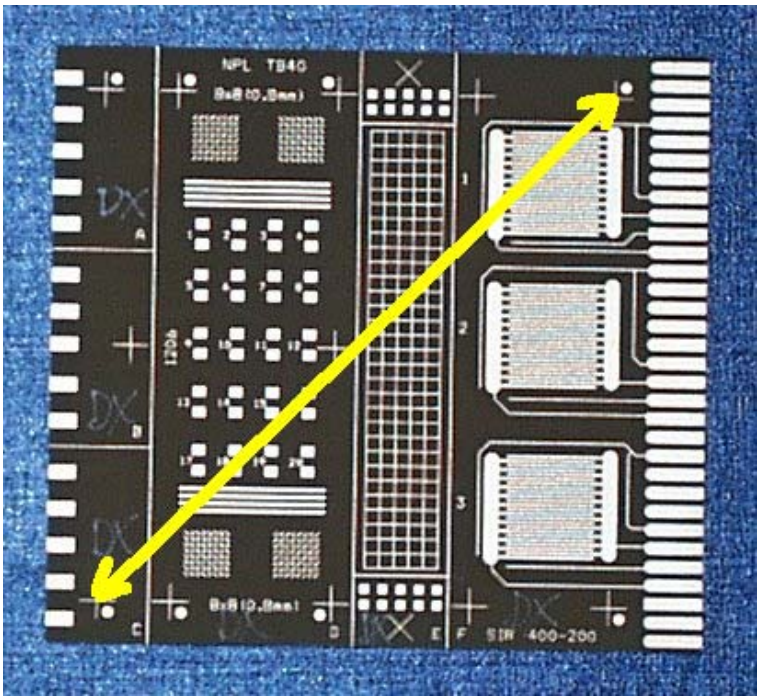

Figure 5: Location of dimensional stability measurement

3.2. Results

The average dimensions and percentage changes after 2 and 4 lead-free profiles are given in Table 2. There were minimal changes in the measured dimensions.

Table 2: Dimensional stability results after lead-free conditioning

	As received	After 2 lead-free Profiles	After 4 lead-free profiles	% change
BT Epoxy	99.10	99.13	99.08	0.0002
FR4	99.12	99.15	99.12	0.0000
Polyimide	99.11	98.92	98.86	0.0025
High Tg Epoxy	99.25	99.08	99.06	0.0019

4. BEND TESTING AT ELEVATED TEMPERATURES

4.1. Procedure

As-received samples of the four laminate types were tested for resistance to bend at elevated temperatures. Coupon E, as highlighted in Figure 6 was guillotined from the main PCB and placed in a special jig, as detailed in Figure 7. In the jig, the coupon was supported at either end and weighted in the centre with a 0.5kg mass. The jig and coupon were then placed in an air circulation oven at 225, 250 or 275^{o}C for 15 minutes. The coupons where then removed and allowed to return to room temperature and the resulting maximum downward deflection was measured using a height gauge.

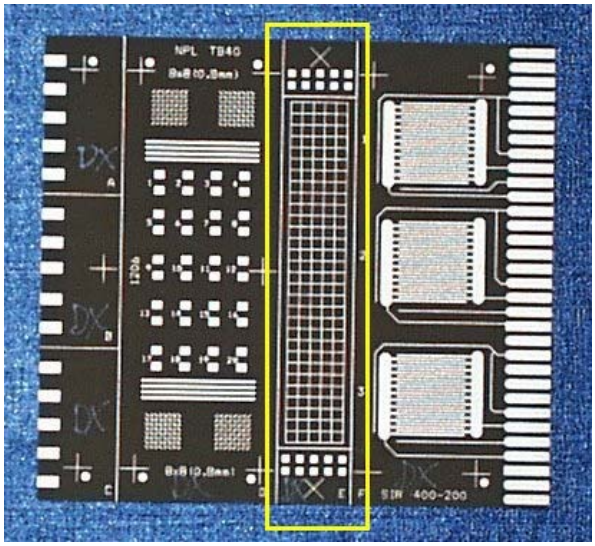

Figure 6: Test coupon for bend testing

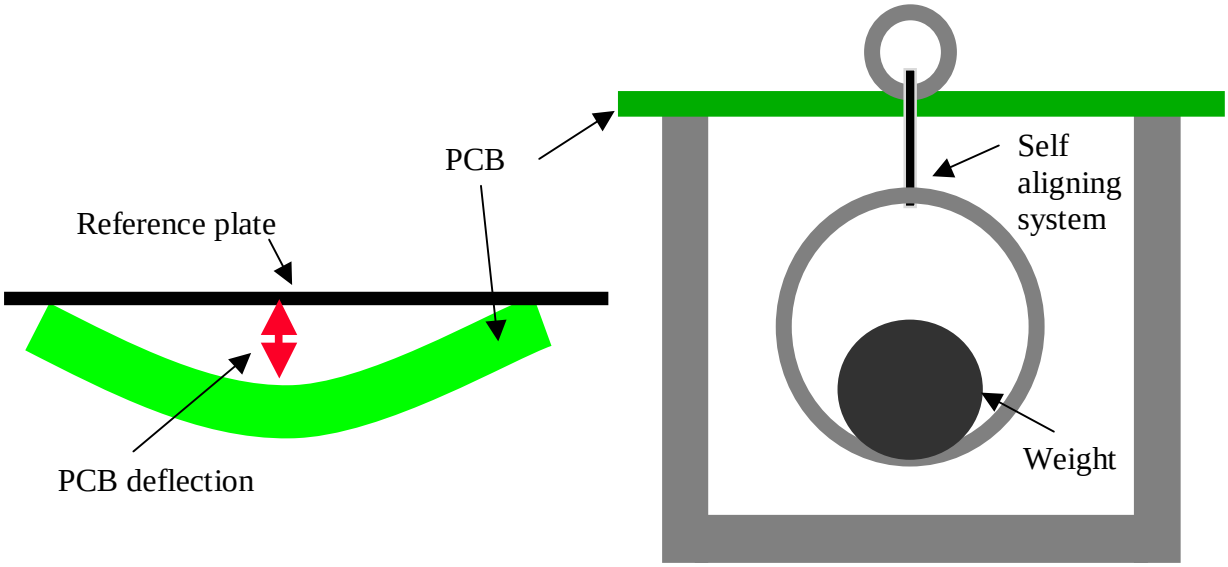

Figure 7: Bend test jig and details of deflection measurement

4.2. Results

The average deflections measured after the elevated temperature soak for the four laminate types are given in Table 3 and shown graphically in Figure 8.

Table 3: Average deflection measurements for four PCB laminate types

Average deflection (mm)	**225 °C**	**250 °C**	**275 °C**
BT Epoxy	0.36	0.28	0.83
FR4	0.73	1.35	6.1
Polyimide	0.03	0.08	0.10
High Tg Epoxy	0.37	0.58	0.74

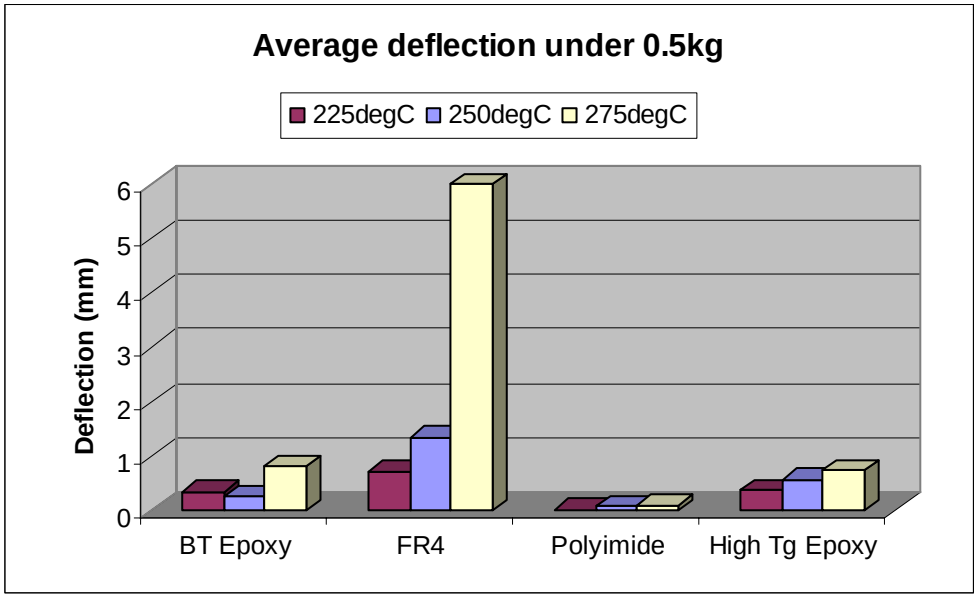

Figure 8: Average deflection of four laminate types after 15 minute soak at elevated temperatures

In all cases, the degree of deflection increased with increased soak temperature. However, there were significant differences in the magnitude of these deflections. The FR4 samples had the least resistance to bend and hence exhibited the greatest deflection - in general greater than five times that exhibited by the other three laminate types. There was relatively little change with soak temperature in the deflection for polyimide laminate, indicating it's suitability as a substrate for lead-free soldering technologies. The BT epoxy laminate showed little change between the deflections at 225 and 250^{o}C, but did show an increase in deflection at 275^{o}C indicating that the laminate would need to be used with care for lead-free soldering, particularly if the reflow profile is expected to be hotter. High Tg epoxy laminate showed increased deflection with increased soak temperature. It did not perform as well as the BT epoxy laminate at 225 and 250^{o}C but performed similarly for a soak temperature of 275^{o}C.

5. SURFACE INSULATION RESISTANCE TESTING

Surface insulation resistance testing was under taken on samples of each laminate type to determine the effect of higher temperature processing on the likely reliability of substrates manufactured from these laminates.

5.1. Procedure

Twelve combinations of coupon material and ageing process were examined using surface insulation resistance (SIR) measurements to evaluate the effect of coupon material and ageing process on reliability of PCBs.

The SIR measurement was performed under controlled temperature and humidity at 85°C/85%RH respectively. A bias of 50V DC was applied during a test period of seven days. The SIR was measured every 15 minutes, and three test SIR patterns were measured for each combination. The results were averaged from the three test patterns.

5.2. SIR measurement results

The average log SIR results with time for the 12 combinations were plotted in Figures 9 to 12. The samples were coded and conditioned as shown in Table 4, and the conditioning was the same as that used for the solderability testing.

Table 4: Sample conditioning

Sample Code	Laminate	Conditioning process
AA	BT epoxy	As-received
AB		4 SnPb profiles
AC		4 lead-free profiles
BA	FR4	As-received
BB		4 SnPb profiles
BC		4 lead-free profiles
CA	Polyimide	As-received
CB		4 SnPb profiles
CC		4 lead-free profiles
DA	High Tg epoxy	As-received
DB		4 SnPb profiles
DC		4 lead-free profiles

From Figures 9 to 12 it can clearly be seen that final SIR value did not show any marked differences due to the different conditioning profiles. However, there were some differences between the laminate types. The final SIR values for the BT epoxy, FR4 and polyimide coupons were similar, but were about one decade lower for the high Tg epoxy coupon. For BT epoxy and FR4 coupons, the conditioning profiles improved the SIR results at the beginning of the tests. This was probably because the reflow process volatilises the contamination on the coupons, and hence “removes” its contribution to lower initial SIR values. However for high Tg epoxy and polyimide coupons the ageing process did not show this cleaning function.

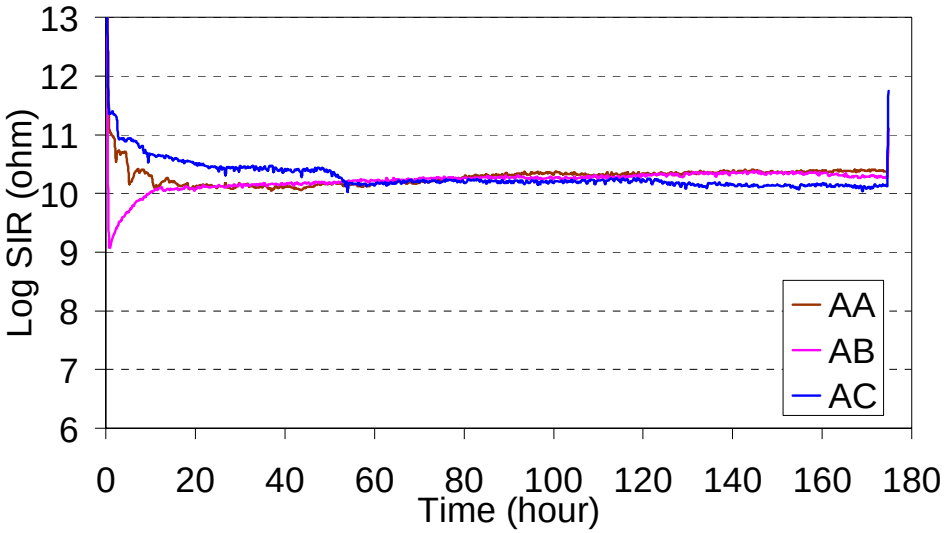

Figure 9: SIR results for BT epoxy laminate

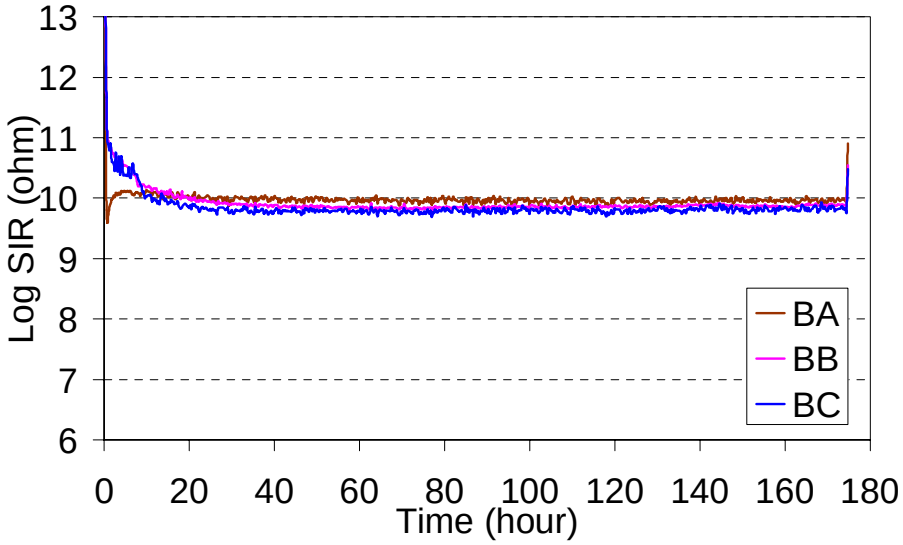

Figure 10: SIR results for FR4 laminate

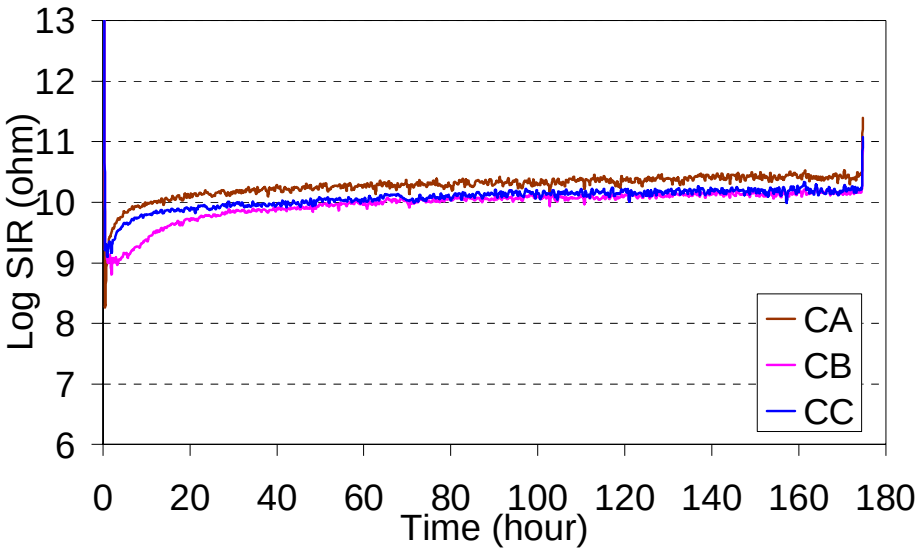

Figure 11: SIR results for Polyimide laminate

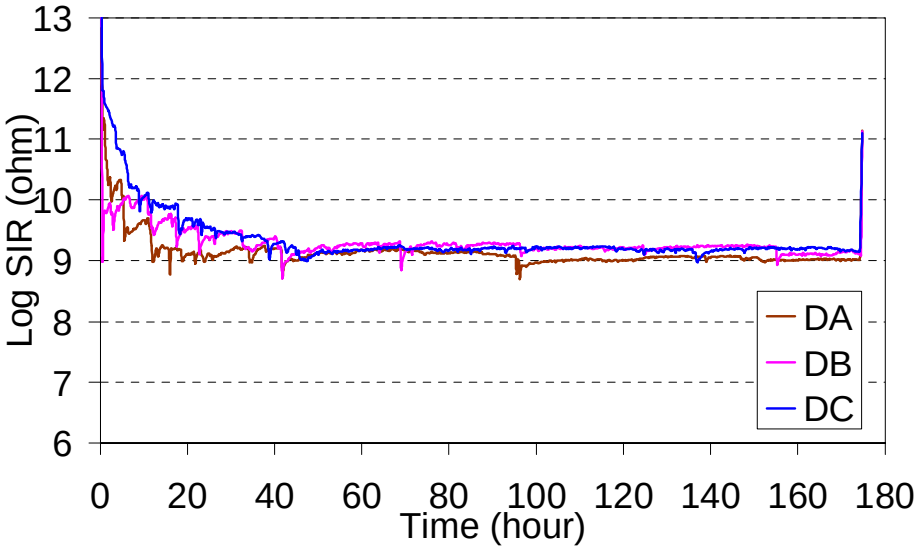

Figure 12 : SIR results for high Tg epoxy laminate

5.3. Discussion

PCB laminate material can have an effect on reliability of electronic printed circuits. BT epoxy, FR4 and polyimide laminates showed higher SIR values than did the high Tg epoxy laminates, and therefore should have higher reliability for lead-free applications. Reflow processing can improve the reliability of BT epoxy and FR4 laminates.

6. AUGER SURFACE ANALYSIS

The higher temperature profile associated with lead-free soldering is speculated of causing degradation of the surface finish on the pads of a PCB. This degradation may be affected by the PCB laminate material, and hence as well as determining the process effects due to high temperature profiling, the surface composition after reflow has been investigated. Hence, a possible relationship between surface finish and process effects has been investigated. Such a relationship would be useful in understanding the behaviour of the material and in ameliorating any deleterious effects.

The determination of the surface finish on the NiAu pads for the various PCB materials was achieved using Auger Electron Spectroscopy (AES), following conditioning as detailed in Section 2.3.

6.1. Procedure

A Varian AES system was used to carry out the investigation. The vacuum system had a base pressure of 10^{-7} Pa, and a 5keV 5μA electron beam was used. The analysis was performed in the differential mode using 5V modulation. Peak to peak intensities were corrected using sensitivity factors from the Handbook of AES from Physical Electronics *(published by Physical Electronics, Inc. 6509 Flying cloud Drive, Minnesota 55344, USA)*. The surrounding pad areas were covered with aluminium foil to prevent charging. The samples were gently sputtered with a 2keV argon beam to remove adventitious carbon contamination. Following acquisition of the data, the sensitivity factors were applied, and the results normalised after removing the carbon peak.

6.2. Results

The normalised AES results for NiAu finished pads from the four substrates are presented in Figures 13 to 16 for BT epoxy, standard FR4, polyimide, and high Tg epoxy materials, respectively.

The results are presented with Au in the top part of the column, and as can be seen for each PCB type the surface finish does not comprise solely gold, other elements are present, in some cases in significant quantities.

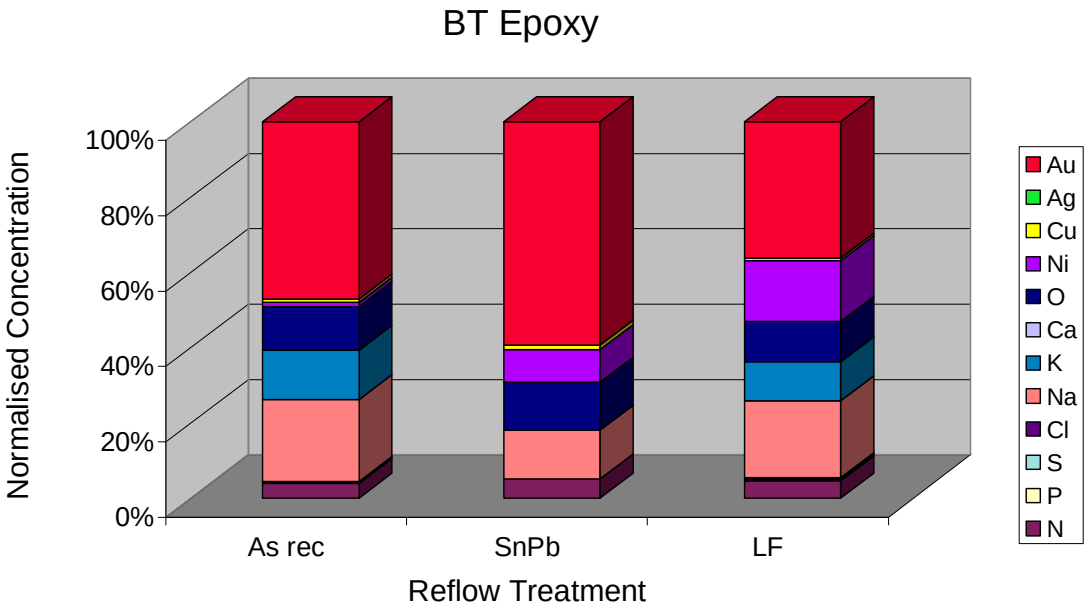

Figure 13: Normalised AES results for BT epoxy

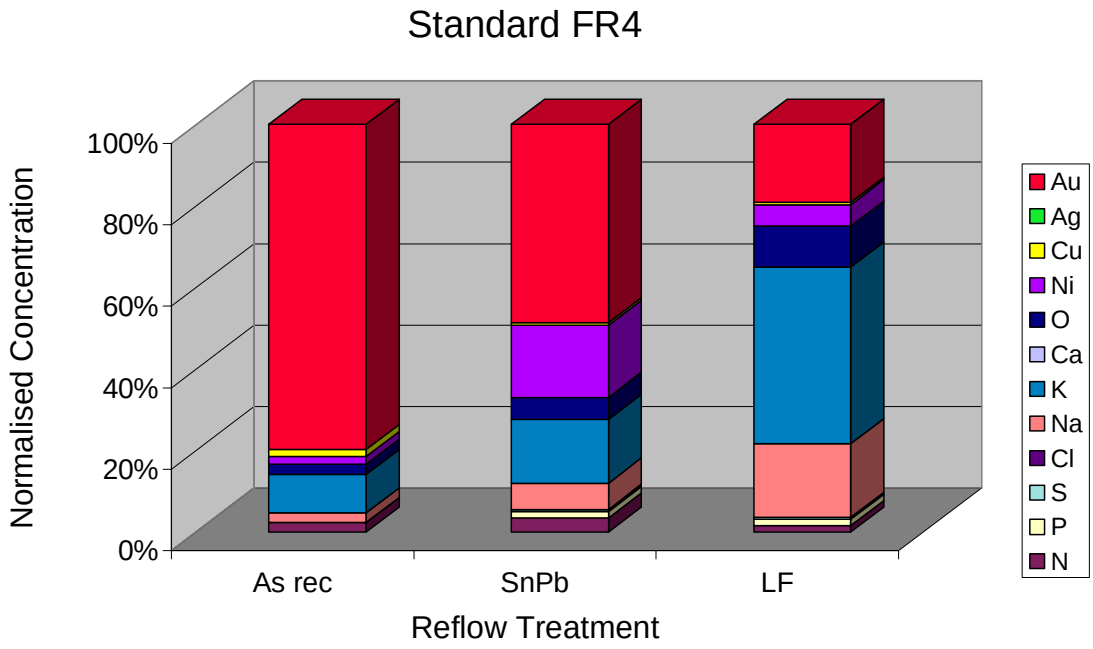

Figure 14: Normalised AES results for FR4

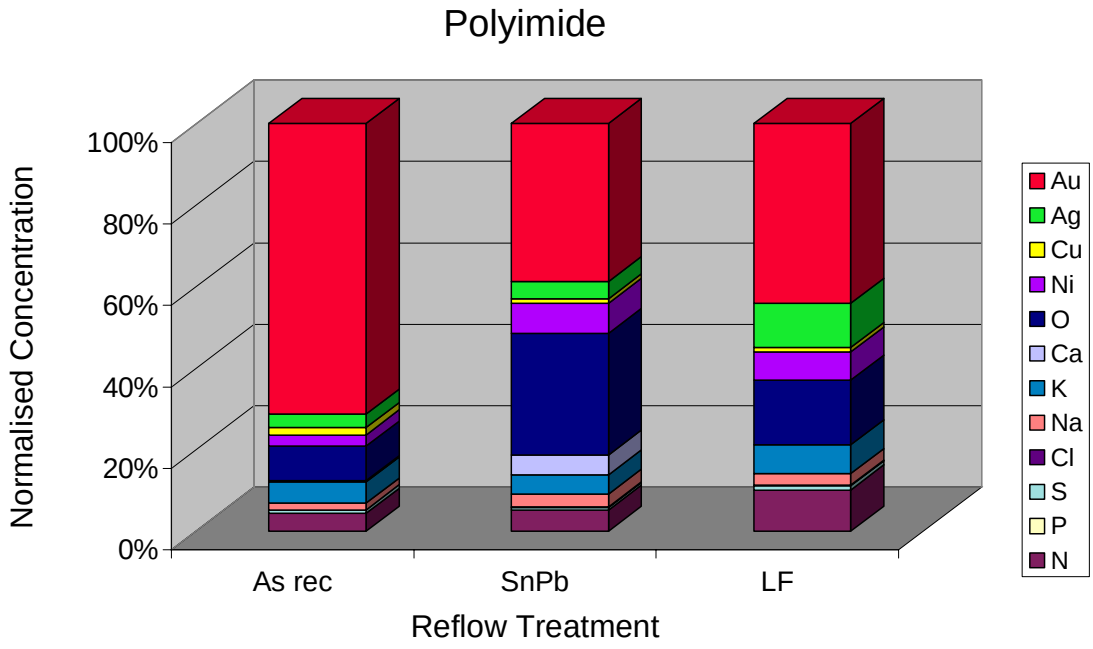

Figure 15: Normalised AES results for polyimide

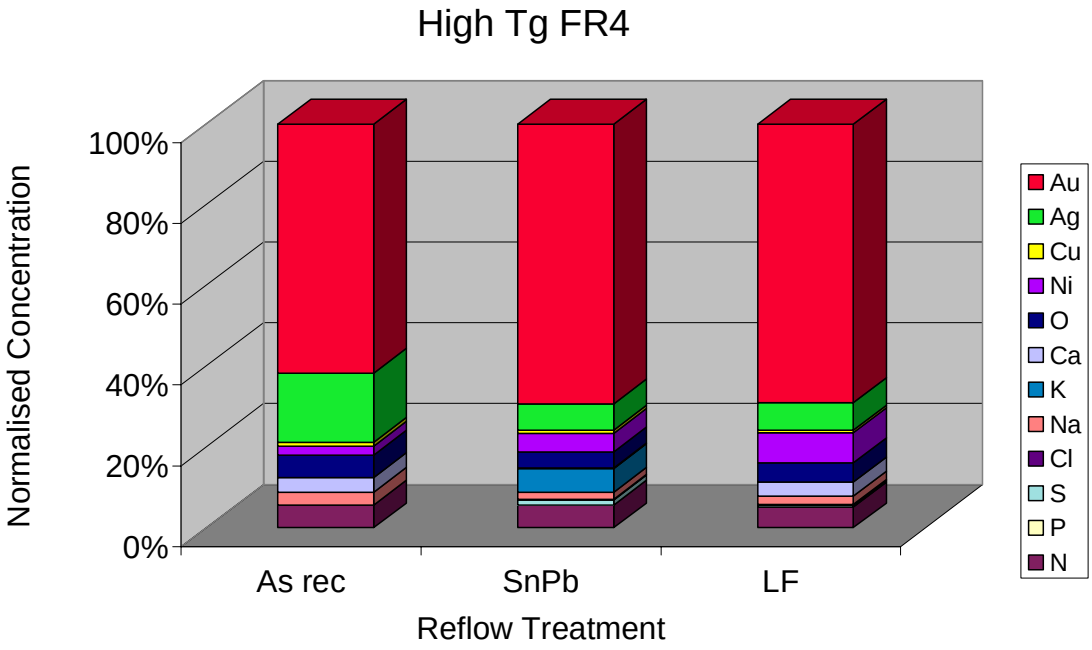

Figure 16: Normalised AES results for high Tg epoxy

6.3. Discussion

There are three observations to make on the occurrence of elements other than Au:

- There are a number of elements that occur at the surface that are probably only there in trace concentrations, but none the less are significant at the surface. These probably derive from the plating chemistries or from adventitious contamination. The reactive alkali metals Na, K and Ca and the halide Cl probably originate from the plating chemistries. S and P probably arise from the plating chemistry, but they may be adventitious, and in any event are always in very low concentration. The occurrence of N is not usually seen as an adventitious surface addition, and hence again presumably comes from the plating chemistries. A typical process line and the chemistries associated with each stage are given in Table 5. The Table shows that there is a wide range of chemistries, which can be potential sources of the contamination seen on the surface. Of particular note is the potassium gold cyanide used in the immersion gold process, and is an obvious source of alkali metals, and possibly the nitrogen.
- The occurrence of Ni at the surface is not unusual. With a thin layer of Au, typically 0.1µm or less, the diffusion of Ni through thin Au layer can occur and will increase with additional heating. This will reduce the solderability of the Au surface. The Ni / Au ratio always increased with increasing profiling. Copper may also appear at the surface via some diffusion route from the underlying pad, but the level is minimal and of no consequence.
- The presence of Ag on the polyimide and high Tg epoxy laminates is interesting. It is probably a contaminant from the plating processes but unlikely to have any effect on the solder processing of the board.

Table 5: Typical Bath Concentrations for the Electroless Nickel/Immersion Gold Technology (Reference 3)

Bath	**Chemicals**	**Concentration in Bath (g/l)**
Cleaner	Phosphoric acid Sulfuric acid Hydrochloric acid Alkylphenolpolyethoxyethanol *Two other confidential chemicals	50.8 138.0 17.9 18.0
Microetch	Sodium hydroxide Hydrogen peroxide Copper sulfate pentahydrate Sulfuric acid *Two other confidential chemicals	0.17 35.88 45.00 87.40
Catalyst	Hydrochloric acid *Four other confidential chemicals	55.80
Acid Dip	*Two confidential chemicals	
Electroless Nickel	Nickel sulfate *13 other confidential chemicals	37.24
Immersion Gold	Potassium gold cyanide *Four other confidential chemicals	3.00

Regarding the PCB materials the results fell into into two groups: one where the gold remained approximately constant, and the other where the gold level dropped with reflow heating.

- First group. Both BT epoxy and high Tg epoxy laminates showed this behaviour. For the high Tg epoxy material the Au level was high at ~70%, with no other element present in significant quantities. But for the BT epoxy the Au level was lower ~50% and there was significant amount of alkali metals at the surface.
- Second group. The FR4, and to a lesser extent the polyimide, showed reducing levels Au with heat treatment. For the FR4 the reduction in Au was primarily due to the increase in K and Ca, with the Ni and O also increasing. The large increase in the alkali metals may be attributed to the plating chemistries, but since the same plating line was used for all finishes, the laminate itself must be implicated in contributing to the appearance of these species. The laminate probably itself does not contain alkali metals, but they could have been absorbed during manufacture, and then released through diffusion during the subsequent reflow thermal cycles.

7. GLOBULE SOLDERABILITY MEASUREMENT

7.1. Procedure

The test samples consisted of 4 X 2 mm copper tracks with NiAu finish on four material coupons, which were BT epoxy, FR4, polyimide and high Tg epoxy. The test samples and aging conditions were designated by sample code, as shown previously in Table 4.

The solderability test was carried out using a Multicore MUST II. The test conditions were in accordance with the IEC standard 68-2-69, and the instrument was used in the globule mode, using a 95.5/3.8/0.7 SnAgCu solder. A 200 mg pellet was used and the solder temperature was 270°C. The flux used was pure rosin with 0.5% halide, in accordance with IEC 68-2-20 and was purchased from Multicore Solder as Actiec5. The immersion speed was 1mm/s, the immersion depth 0.1mm, and no pre-heat was used. The force data were acquired over a 5 second period. The pads were dipped at an angle of 45°. Six pads on two coupons for each laminate and ageing condition were tested. The test results given are an average of the values for these 6 pads.

7.2. Globule solderability measurement results

Wetting force and wetting time data for the 12 combinations are presented in Figures 17 and 18. It can be seen from these Figures that for both the force at 2 seconds and the time to 2/3 maximum force, there was little difference among the laminate materials. However, there was a significant difference between different ageing processes. In general as-received samples gave the highest wetting force and shortest wetting time, and the samples aged by the lead-free reflow process, showed the lowest wetting force and longest wetting time. *This suggested that the solderability of the solder pad was not affected by laminate material, but dramatically influenced by the conditioning process*. The higher temperature reflow profile

associated with lead-free soldering significantly decreased the solderability even further, (i.e. for combinations AC, BC, CC and DC).

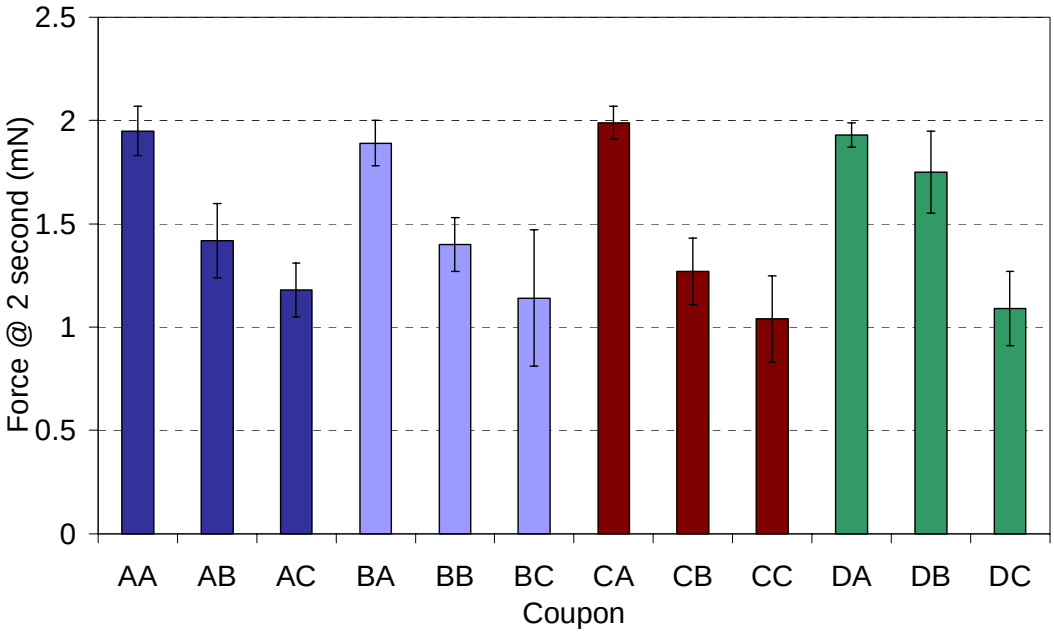

Figure 17: Solderability results (average force @ 2 seconds) for the four laminate types

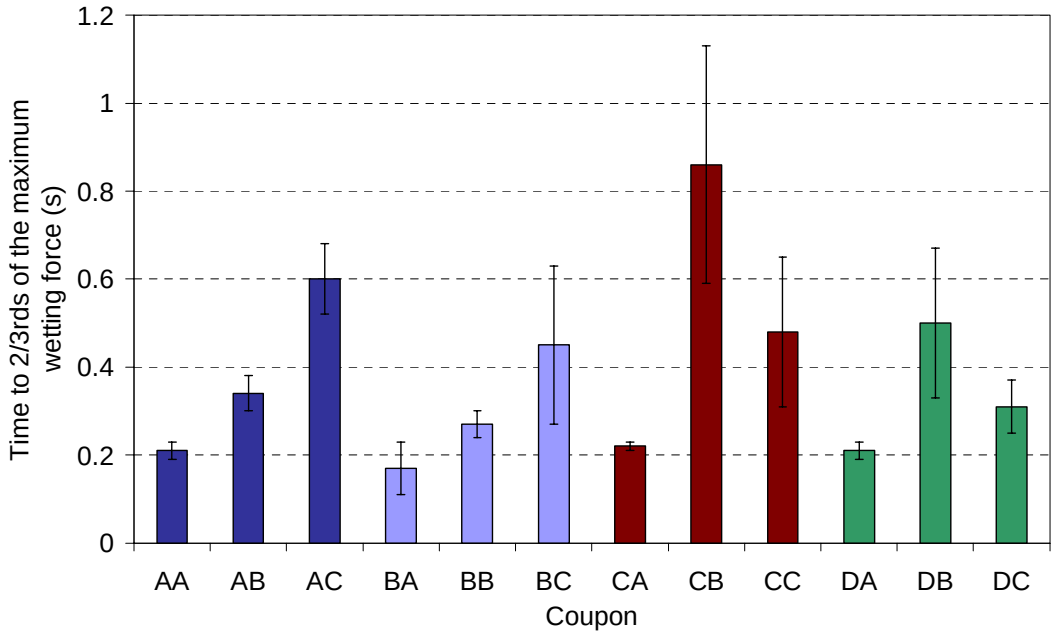

Figure 18: Solderability results (average time to 2/3 of maximum wetting force) for the four laminate types

8. SOLDERABILITY DOT TESTING

8.1. Procedure

The procedure was simialr to that used for globule solderability testing, coupons of all four laminate types were tested under the same three conditioning profiles, as-received, 4 SnPb profiles and 4 lead-free profiles.

A mini stencil was used to print solder paste onto the tracks on coupons D. The paste used was no-clean type 3 rosin paste, and the stencil contained 72, 0.5 X 0.5 mm square apertures in 4 rows, 18 apertures on each row, as shown in Figure 19. The gap between the apertures increased from 0.16 to 0.97 mm in 0.05 mm increments. The stencil thickness was 100µm. Two coupons were printed and reflowed for each combination. Examples of solder paste dots after printing and reflow are shown in Figures 20 and 21. The number of dots that did not coalesce after reflow was counted for each board, and the results were averaged over two boards or six strips.

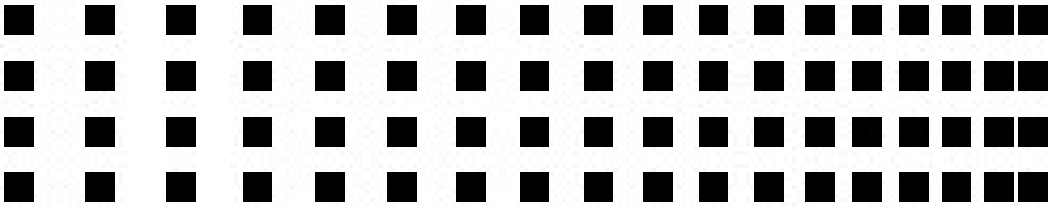

Figure 19: Dot test stencil aperture array

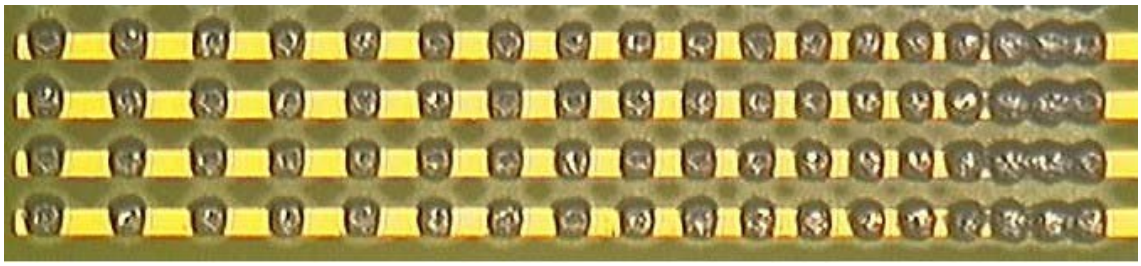

Figure 20: Paste dots after printing

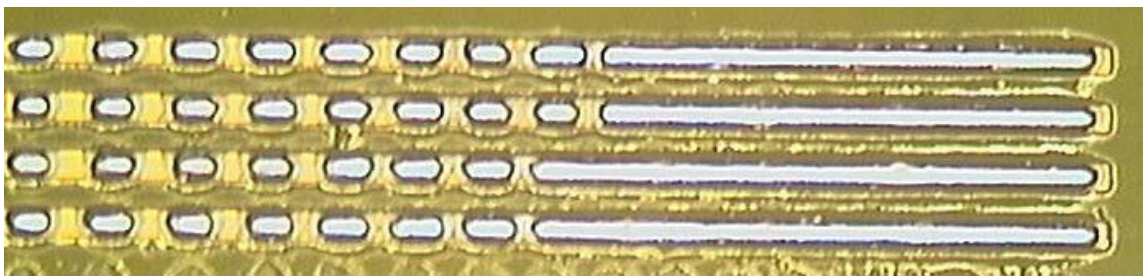

Figure 21: Paste dots after reflow

8.2. Dot test results

The average number of uncoalesced dots and the standard deviation for the 12 combinations of laminate material and conditioning are presented in Figure 22. The sample coding was that given in Table 4.

It can be seen from Figure 22 that there was little difference among the different laminate materials. However, there was a significant difference between different conditioning processes. Not surprisingly the as-received samples gave the lowest uncoalesced dot value, and the sample aged by lead-free reflow process showed the highest uncoalesced dot value. ***This suggested that the solderability of solder pad was less affected by coupon material, but dramatically influenced by the conditioning process.*** Furthermore the higher temperature reflow profile associated with lead-free soldering significantly decreased the solderability (i.e. for combinations AC, BC, CC and DC).

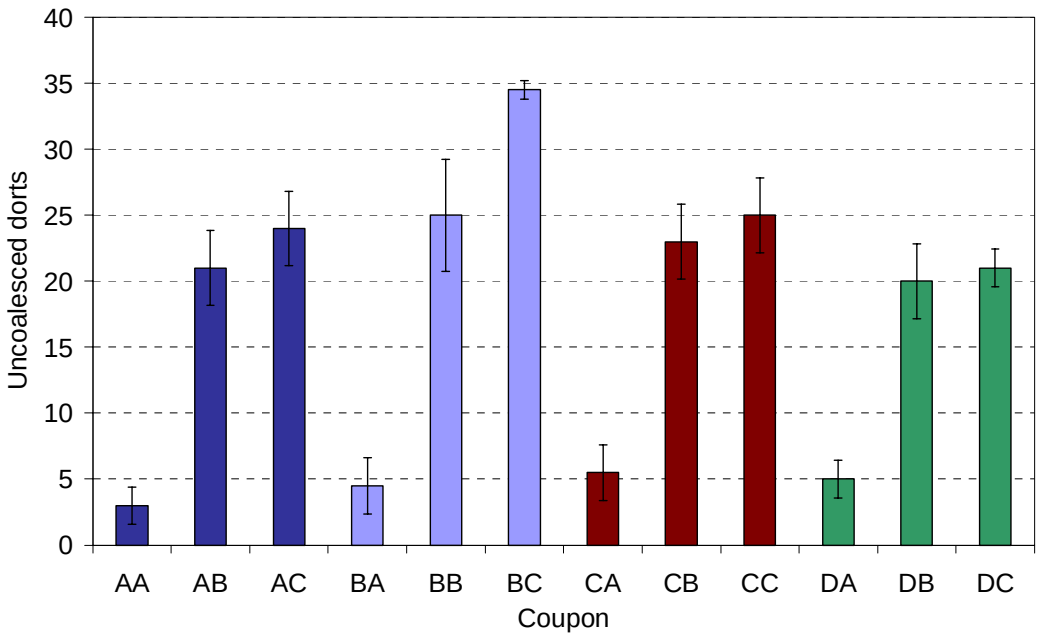

Figure 22: Uncoalesced dots values for different laminate types and ageing conditions

9. Conclusions

Five different aspects of PCB manufacture and processing have been investigated to explore whether the move to the hotter reflow profiles associated with lead-free soldering would affect the performance of four common laminate types (i.e. BT epoxy; FR4; polyimide; high Tg epoxy). Boards of the different laminates were subjected to multiple cycles of temperature profiles representative of lead-free soldering, and then studied in relation to dimensional stability, bend testing, SIR, surface composition and solderability. The salient conclusions of this study were:

- The increased temperatures associated with lead-free soldering had little, or no, effect on dimensional stability or SIR of the boards, and did not vary with laminate types.

- As expected, the higher Tg laminate exhibited the best (most stable) behaviour in the bend tests at the higher temperatures

- FR4 laminate subjected to loading during lead-free reflow soldering might become warped during the soldering operation. Such loading might arise, say, from an uneven multilayer build, or a double-sided assembly supported only in the four corners, whilst travelling on a mesh belt.

- The poor SIR performance of the high Tg epoxy laminate is also a cause for concern, with regard to its potential use in lead-free soldered products. More work needs to be undertaken to ascertain whether or not this is a more widespread phenomenon.

- The FR4 laminate, and to a lesser extent the polyimide material, exhibited a worrying reduction in solderability after being subjected to multiple lead-free soldering profiles. This effect was more marked for the dot testing than for the globule testing

- The solderability of the solder pads appeared to be independent of laminate material, but it was dramatically influenced by the conditioning process. The increased ageing associated with the lead-free profiling (compared with that of SnPb profiling) caused a significant reduction in solderability of all the laminates. These results imply that if standard immersion gold finishes are used for lead-free assembly (where multiple profiling will occur – e.g. with double-sided soldering), then lower yields can be expected on the second side reflow compared with those currently achieved with SnPb double-sided soldering.

The work has highlighted two areas of concern if current laminate types and immersion gold pcb finishes are used in lead-free soldering technologies.

A. If a board is going to be subjected to stresses during the lead-free soldering operation, then manufacturers wishing to have a flat, or near flat, assembly after soldering, should consider whether or not the Tg of the base laminate material is appropriate.

B. The lower soldering yields after more than a single lead-free soldering operation, suggests that more aggressive fluxes must be used to maintain soldering yield.

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