Controlling Moisture during Inner layer Processing.

John A. Marshall
MacDermid Inc.
Waterbury, CT

Abstract
The conversion to higher temperature "Lead Free" assembly reflow conditions has created an increased awareness that entrapped or absorbed moisture is a frequent root cause of thermally induced delamination at assembly reflow. There are two connected failure modes from entrapped moisture; incomplete resin cross-linking resulting in premature resin decomposition and also severe Z axis expansion from "explosive vaporization of the entrapped moisture at elevated temperatures at assembly reflow". Ultimately, both result in delamination failure.

Other papers have shown the negative effects of entrapped moisture before lamination including delamination, red color, reduced thermal reliability and increased high speed signal loss. In this paper, various materials were tested for moisture sensitivity during lamination. Tests were performed at varying lamination conditions including a pre-vacuum step and "kiss" step. Pressure and cure temperature parameters were evaluated for minimizing or eliminating the effect of trapped moisture. Also included are the results of inner layer moisture removal baking conditions and their effect on peel strength and thermal reliability.

Introduction
The overall reliability of a finished Multilayer Printed Circuit Board is greatly affected by trapped moisture. In severe cases, moisture interferes with the resin curing during lamination and the effects are clearly visible as red color, dry glass or blisters after outer layer etch. Moisture can react with components in the flame retardant package, releasing corrosive brominated volatiles during lamination that will often result in a red color of the Oxide Alternative coating. (Fig 1) Additionally, trapped moisture increases resin flow which can result in a "dry glass" or "resin starvation" condition when insufficient resin remains in the multilayer package to fully encapsulate the glass bundles. (Fig 2, Fig 3). Resin squeeze-out is also increased and frequently is a darker color bead with air bubbles. (Fig 4) Both of these conditions are often found around the perimeter of the laminated panel and around tooling pin holes where the moisture is collected as it is forced outwards during lamination. In severe cases, red color, dry glass and delamination occur during lamination, becoming visible after outer layer etch. Another common failure site is in plated sub assemblies with high hole density, indicating the possibility of moisture incursion after drilling and increased heat retention due to increased amount copper mass of the plated through holes (Fig. 5). With less severe cases, the effects of trapped moisture are not clearly evident until later in the process and, in the case of non HASL PCBs, may not be visible until after reliability testing or assembly reflow. (Fig 6). During high temperature Lead-Free Reflow, the expanding moisture can result in explosive forces that cause severe delamination failure. Delamination during assembly reflow from entrapped moisture can occur without initially seeing red color change, depending on the laminate material composition.

In many cases, Phenolic cured FR4 materials are replacing the traditional DICY cured materials. Also, the use of high performance/high speed materials continues to increase. With this trend we have seen a reduction in the incidence of Red Color after lamination, but trapped moisture continues to cause reliability issues with a variety of non DICY materials. In one of these cases, with a Phenolic 180 Tg material, we saw red color only at elevated lamination temperatures without a "Pre-vacuum/kiss step". However, we saw premature delamination after Lead Free Reflow with all lamination conditions tested when moisture was introduced. The volatiles released during resin decomposition are corrosive and result in the oxide alternative changing from brown to red as a result of the acidic volatiles dissolving the oxide alternative coating. (1) Halogen-Free laminate materials are typically not as susceptible to red color change as halogenated materials, but are still moisture sensitive.

In addition to red color problem, other well documented effects of trapped moisture include reduced thermal and electrical properties including lower Tg, Td and T-288, increased CAF growth, a reduction in switching speeds and increase in propagation delay times. (2) (3) (4) (5)

Prepreg Moisture
Before lamination, moisture can be present in the prepreg itself, primarily from poor, high humidity storage conditions especially with extended "open bag" times. Transporting open bags of pre-preg from cold storage through an uncontrolled environment can lead to rapid moisture condensation on the prepreg. Any opened prepreg packages must be re-sealed and

have fresh desiccant placed inside before returning to cold storage. Allowing time for the prepreg packages to equilibrate to lay-up room conditions before opening is critical in preventing condensation.

Increased awareness of the potential problems caused by trapped moisture in the prepreg has prompted many PCB Fabricators to improve their pre-preg storage conditions and general handling of moisture sensitive materials. PCB Laminate Suppliers have detailed handling, storage and lamination recommended SOPs specifically intended to prevent trapped moisture problems. The use of prepreg “dry boxes” and vacuum desiccation, immediately before lay-up, prevents trapped moisture problems with moisture sensitive materials.

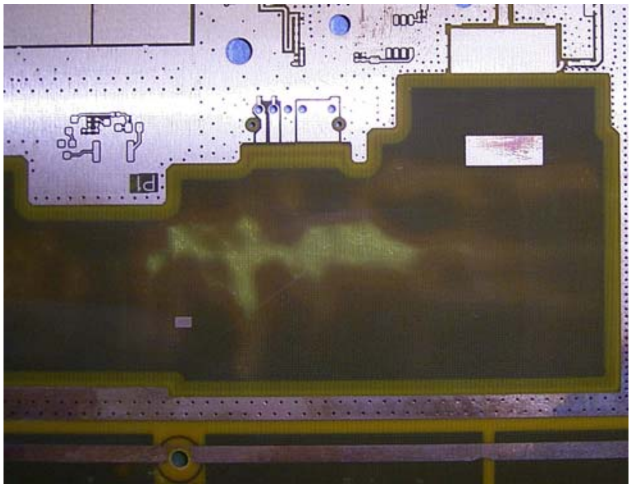

Fig. 1 - Red Color & Dry Glass

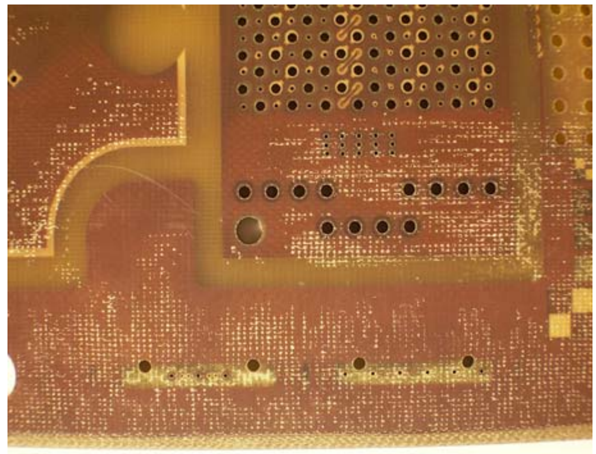

Fig. 2 - Red Color & Dry Glass

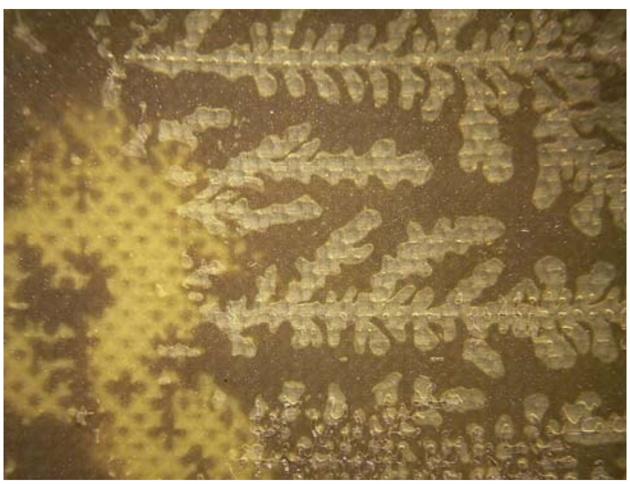

Fig. 3 – Dry Glass

Fig. 4 - Resin Bead with Moisture

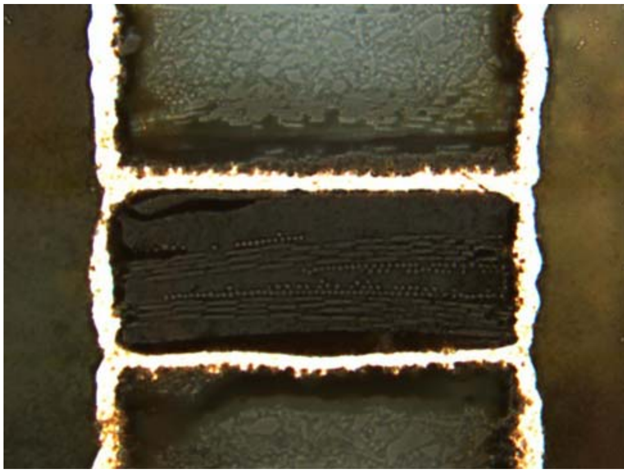

Fig. 5 – Delamination After Reflow

Fig. 6 – Delamination After Reflow

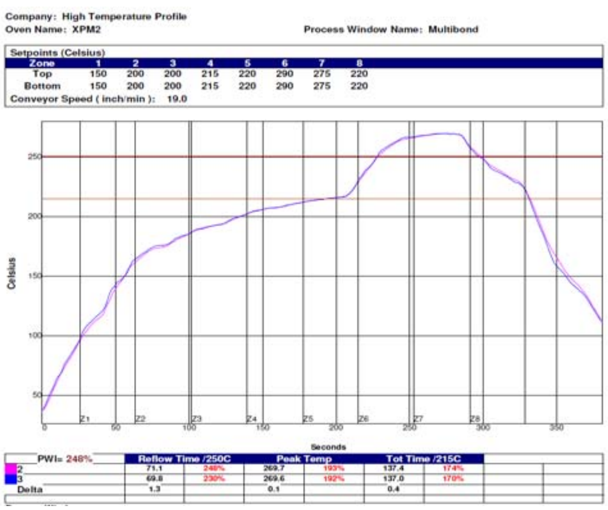

Fig. 7 – Lead-Free Reflow Cycle

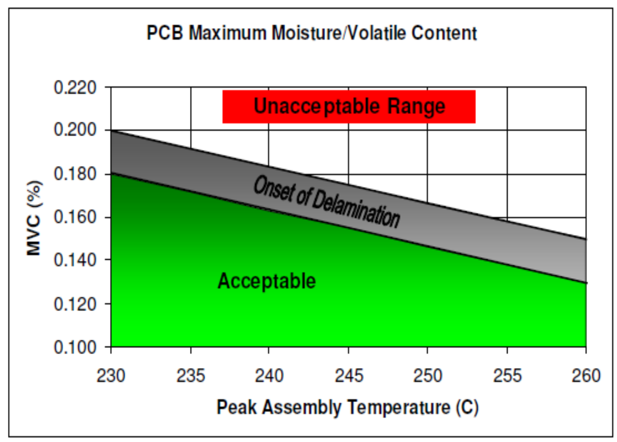

Fig. 8 – Maximum Moisture

Inner Layer Moisture

Moisture can also be absorbed and trapped in the inner layer cores before lamination from extended hold time in an uncontrolled, high humidity environment following DES, before lamination. Many "high performance" materials can be especially sensitive to trapped moisture. Different laminate materials have varying degrees of moisture uptake through processing and storage and must be handled accordingly. Laminate Suppliers "Best Practice" recommendations are to maintain inner layer moisture content at < 0.15% (Fig. 8), include rack baking before lamination, for 30 minutes minimum at 100^0C, especially if the finished Multilayer PCB is intended for high temperature lead free reflow. (6)

Many high volume PCB manufacturers have retrofitted additional drying modules to their existing horizontal oxide alternative lines in order to improve their drying effectiveness. There has been a dramatic increase in the number of PCB fabricators that utilize an oven bake after Oxide Alternative, before lamination. Typical baking conditions would be 30 to 60 minutes at 120^0C with inner layers slip-sheeted with paper in < 1 inch stacks.

Moisture Sensitivity Test Results

Initial moisture sensitivity testing had focused on Dicy cured materials as they were seen as a group to be most sensitive to red color change and delamination from trapped moisture. However, customer experience with Non-Dicy cured materials has shown similar delamination problems and, to a lesser extent, red color change from trapped moisture.

Tests were run on various materials to determine their overall moisture sensitivity, both to red color during lamination and Reflow Cycles to Delamination. Solid copper 9" X 9" inner layers were run through horizontal Oxide Alternative process, followed by vacuum lamination with 0.05 ml DI water introduced between the prepreg and oxide alternative coating.

Samples were laminated with and without a “Pre-vacuum/kiss step” as the first step in the lamination cycle in order to evaluate it’s effectiveness in removing moisture from the multilayer packages. The tests evaluated their impact on Red Color and the number of Reflow Cycles To Delamination using 6 prepreg materials. A high temperature Lead Free Reflow profile was used to test “worst case” reflow conditions, with Peak Temperature of 270^0C and 70 seconds > 250^0C (Fig 7). The “Pre-vacuum/kiss step” followed typical laminate suppliers’ recommendation for optimizing moisture removal. Initially, full vacuum pressure of 28 in/Hg was applied for 20 minutes before closing the platens and starting the press cycle, to aid in moisture removal from the multilayer package. The first step in the lamination cycle was at 40^0C, for 30 minutes, with full vacuum (28 in/Hg) and low (5psi) platen pressure. The following materials were evaluated for moisture sensitivity and the effect of a “Pre-vacuum/kiss step”. Sensitivity was determined by any red color change and second by change in Lead-Free Reflow cycles to delamination performance. Each material type was laminated using the manufacturers recommended press cycle.

Table 1 - Materials Tested

Material	Type	Tg	Td	Moisture Absorption
1	Halogen Free	150^0C	320^0C	0.06%
2	Phenolic FR 4	180^0C	340^0C	0.15%
3	High Performance	170^0C	340^0C	0.15%
4	Multifunctional FR 4	200^0C	360^0C	0.06%
5	Low Loss	180^0C	360^0C	0.06%
6	High Speed	200^0C	360^0C	0.01%

Table 2 - Red Color Sensitivity Results

Material	No Pre-Vacuum, No Kiss	Pre-Vacuum & Kiss
1	NO	NO
2	YES	NO
3	YES	NO
4	YES	NO
5	YES	YES
6	YES	NO

Table 3 - Test Results 270^0C Reflow Cycles to Delamination

	270^0C Reflow Cycles to Delamination		
Material	0.05 H_2O ml	0.05 ml H_2O + Vac/Kiss	No H_2O
1	3	4	6
2	8	9	14
3	>20	>20	>20
4	1	7	>20
5	>20	>20	>20
6	4	6	15

The Results show a wide range of moisture sensitivity amongst the materials tested. The Halogen Free material #1 was the only material tested that did not exhibit Red Color change after lamination. The reflow test conditions were extreme for this specific material with a Td of 320^0C. The results indicated a 50% loss of performance from introduced moisture, with “Pre-vacuum/kiss step” having minimal impact. Samples #2 and # 6 had about a 50% reduction in cycles to Delamination from introduced water and minimal positive impact from using a “Pre-vacuum/kiss step”. Sample # 4 appeared to be the most moisture sensitive material tested, with the largest difference in performance from introduced moisture, going from >20 cycles to delamination after 1 cycle without “kiss step”. Even with the “Pre-vacuum/kiss step”, there was a significant reduction in cycles to delamination, from >20 cycles to 7 cycles. On the other end of the spectrum, materials #3 and #5 appeared to have no significant loss of performance from introduced moisture. Both materials had no delamination with up to 20 high temperature Lead Free Reflow cycles, indicating extremely robust thermal performance. Interestingly, sample #5 was the only sample tested with red color visible after lamination, both with and without “Pre-vacuum/kiss step”; however reflow performance was not significantly affected by the introduced moisture.

Delamination during assembly reflow from entrapped moisture can occur without red color change during lamination, depending on the laminate material composition and press cycle used. Moisture interferes with the curing mechanism resulting in reduced cross-linking, at which point the integrity and survivability of the finished board can be severely compromised. In these cases, baking before reflow will have minimal impact on survivability, with significantly less effect than moisture absorbed during storage of finished PCBs. Entrapped moisture inside a finished multilayer circuit board can be difficult to remove, taking up to 12 hours or longer at 150^0C in order to eliminate the moisture from the board. Storing inner layers in a controlled environment as they move through the process, from DES through lamination will minimize any additional moisture uptake. Baking Inner Layers before lamination and using a "Pre-vacuum/kiss step" in the lamination cycle will help prevent delamination problems resulting from entrapped moisture.

Baking Before Lamination Test Results

Baking Inner Layers steadily becomes SOP for High Tg materials to assure moisture content of < 0.15% before lamination. Some material suppliers are evaluating extended Inner layer baking of up to 4 hours at 150^0C, before lamination. Following the Suppliers guidelines, test layers were prepared to evaluate the effect on peel strength of the baking conditions after Oxide Alternative. Two baking temperatures were selected, 120^0C and 150^0C, with baking times in one hour increments up to 6 hours at each temperature.

1 ounce RTF copper foils were processed through Oxide Alternative "M" followed by convection oven baking with the foils placed Oxide Alternative coating up, with each foil surface exposed to air. Copper foils were vacuum laminated following the manufacturers recommended press cycles with three different prepreg materials from the previous testing;

2 - Phenolic 180^0C Tg
4 - High Performance 200^0C Tg
6 – High Speed 200^0C Tg.

After lamination, one inch platers tape was used to define and etch copper strips for peel testing. Peel Strength was measured using a Diventco Peel Tester after lamination and again after 6X 10 seconds solder dip at 288^0C to evaluate any loss of peel strength performance after extended baking. The Peel Strength results are reported in Pounds per Linear Inch as an average of 5 results from 5 strips. (Table 4, Table 5)

Table 4 - 120^0C Baking Peel Strength Results

Material	0	1 Hr	2 Hrs	3 Hrs	4 Hrs	5 Hrs	6 Hrs
2	6.0	5.8	5.8	5.7	5.5	5.4	5.3
4	5.3	5.2	5	4.8	4.8	4.7	4.6
6	4.4	4.3	4.3	4.1	4.0	3.8	3.8

120^0C Baking Peel Strength % Loss Results

Material	1 Hr	2 Hrs	3 Hrs	4 Hrs	5 Hrs	6 Hrs
2	3.3%	3.3%	5.0%	8.3%	10%	11.6%
4	1.8%	6.0%	9.4%	9.4%	11.3%	13.2%
6	2.3%	2.3%	6.8%	9.0%	13.6%	13.6%

120^0C Baking Peel Strength % Loss Results after 6X 10 sec. @ 288^0C

Material	1 Hr	2 Hrs	3 Hrs	4 Hrs	5 Hrs	6 Hrs
2	1.8%	1.8%	5.5%	7.4%	11.1%	14.8%
4	2.0%	6.1%	6.1%	8.2%	10.2%	12.2%
6	0%	2.4%	4.7%	9.5%	14.3%	14.3%

Table 5 - 150^0C Baking Peel Strength Results

Material	0	1 Hr	2 Hrs	3 Hrs	4 Hrs	5 Hrs	6 Hrs
2	6.0	5.7	5.5	5.2	5	4.9	4.8
4	5.3	4.8	4.6	4.5	4.2	4.0	3.8
6	4.4	4.4	4.3	3.9	3.7	3.6	3.6

150^0C Baking Peel Strength % Loss Results

Material	1 Hr	2 Hrs	3 Hrs	4 Hrs	5 Hrs	6 Hrs
2	5.0%	8.3%	13.3%	16.6	18.3%	20.0%
4	9.4%	13.2%	15.1%	20.7%	24.5%	28.3%
6	0%	2.3%	11.4%	15.9%	18.2%	18.2%

150^0C Baking Peel Strength % Loss Results after 6X 10 sec. @ 288^0C

Material	1 Hr	2 Hrs	3 Hrs	4 Hrs	5 Hrs	6 Hrs
2	1.8%	7.4%	12.9%	16.6%	22.2%	25.9%
4	10.2%	14.3%	18.4%	22.4%	28.6%	28.6%
6	2.4%	2.4%	9.5%	16.6%	14.3%	19.0%

The results indicate that peel strength declines from extended baking, with 150^0C temperature having greater negative impact than 120^0C. Baking for one hour at 120^0C resulted in up to 3.3% loss of peel strength and after two hours, up to 6%. Baking for one hour at 150^0C resulted in up to 10% loss of peel strength and after two hours up to 14%. These results confirm the suitability of laminate suppliers' recommendation to bake inner layers before lamination for 30 to 60 minutes at 100^0C-120^0C for moisture removal, without significant loss of peel strength performance. If there is a requirement to bake inner layers or sub-assemblies for more than one hour at 150^0C, then it is recommended to bake before the Oxide Alternative process.

Baking SBU sub assemblies for `1-3 hours at 150^0C before MultiBond can be beneficial. Typically, SBU delamination problems are on outer-most layers of the sub-assembly from the plated copper surfaces. Extended, high temperature baking will remove any trapped volatiles in the hole fill material and the electrolytic plated copper. Baking sub-assemblies will also "anneal", or re-orient the plated copper crystals making them receptive to sulfuric/peroxide micro-etches and consequently optimize the plated copper surface roughness from the Oxide Alternative coating. Care must be taken after electrolytic copper plating sub-assemblies, to fully rinse and dry them before extended baking in order to prevent severe copper surface oxidation from chemical residues.

Currently, many PCB suppliers are baking inner layers in stacks of < 1.5 cm, with inner layers separated with paper or plastic slip sheets. Rack baking is preferred, but adds additional handling after Oxide Alternative. In-line baking after Oxide Alternative, in a hanging or "wicket" oven would be the preferred alternative for an automated Oxide Alternative Process line.

Many Oxide Alternative Horizontal lines have been modified with additional drying capacity, primarily to improve drying of the much thicker sub-assemblies with holes. Additional baking before lamination is recommended, especially for moisture sensitive materials and plated sub-assemblies. After baking, minimizing hold time to less than 4 hours in a controlled environment of < 21^0C and < 50% Relative Humidity before lamination is also recommended. Many customers go directly from baking into lay-up, with no hold times for Halogen Free and many High-Speed/Low Loss materials. Typically, inner layers will be re-baked when the maximum hold time after baking is exceeded.

Conclusions

Entrapped moisture inside a finished multilayer circuit board can compromise the integrity and thermal reliability of a Multilayer PCB. Unlike absorbed moisture that can occur during storage of finished PCBs, the effects of trapped moisture before lamination are typically not reversible.

Controlled prepreg storage conditions, correct equilibration before opening bags and re-sealing open bags are key factors in controlling moisture in prepreg. Holding inner layers only in a controlled environment as they move through the process, from DES through lamination will minimize any additional moisture uptake in the inner layers. Baking Inner Layers before lamination for 30 to 60 minutes at 100^0C–120^0C and using a "Pre-Vacuum" and "Kiss Step" in the lamination cycle will help prevent delamination problems with Multilayer Printed Circuit Boards resulting from entrapped moisture during manufacture.

References

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