Characterization of the Thermal Stability of Electrical Laminates Suitable for Lead-Free Soldering

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Abstract

Lead-free soldering is expected to become the new standard in the future. Different legislations or draft directives target the restriction or the ban of the use of lead in the world. As an example, the European Union adopted the Restriction of Hazardous Substances (RoHS) Directive in January 2003. It will come into effect on July 1, 2006. Most of the lead-free solder alloys melt at higher temperatures than that of the eutectic SnPb solder. The change to higher temperature solder alloys will directly affect the temperature profiles for reflow soldering, wave soldering, rework and repair. Typical lead-free reflow profiles will reach peak temperature of 245°C to 265°C for up to a minute. De-soldering, rework and repair will reach peak temperature above 300°C for a few seconds. In parallel, the complexity of the boards is increasing, leading to thicker multilayer structures. Laminates will thus be submitted to higher temperature for longer time through multiple reflow cycles. It is critical to understand how these new technical requirements will have an impact on the thermal resistance of electrical laminates.

This paper aims to provide correlations between different techniques used to characterize the thermal stability of electrical laminates suitable for lead-free soldering.

- Thermo-Gravimetry Analysis (TGA) was used to measure the degradation temperature (Td) and the time to degradation at a given temperature (D-260, D-288, D-300);
- Thermo-Mechanical Analysis (TMA) was used to measure the time to delamination at a given temperature (T-260, T-288, T-300), the number of temperature cycles before delamination (Nd), and the coefficient of thermal expansion along the z-axis (CTE);
- Differential Scanning Calorimetry (DSC) was used to measure the glass transition temperature (Tg).

The thermal stability data of various epoxy systems will be described and correlated to specific applications needs. Various examples of epoxy systems will be chosen within the portfolio of The Dow Chemical Company.

Results suggest that conventional FR-4 resins might still be suitable for standard FR-4 applications that need only a few lead-free reflow cycles. When the number of cycles increases, enhanced resin systems must be considered to avoid in-process failure. Highly thermo-resistant products are suitable for complex multilayer build-up or for applications targeting high in-use temperature. In addition to thermal stability, other key laminate parameters for board reliability are adhesion and toughness.

Introduction

Lead-free soldering is expected to become the new standard in the future. Different reasons explain this trend. First, different legislations or draft directives target the restriction or the ban of the use of lead in the world. The major ones are the Waste of Electrical and Electronic Equipments (WEEE) and the Restriction of Hazardous Substances (RoHS) Directives in the European Union (EU). The initial efforts of reducing the content of specific hazardous substances in the waste stream were not sufficient. The EU has decided to further restrict the incorporation of these hazardous substances in electronic products by enacting the RoHS Directive. From July 1, 2006, products which do not comply with these directives cannot be placed on the EU market. For instance, no more than 0.1% of lead shall be tolerated by weight of homogeneous material, e.g. which cannot be mechanically disjointed into different materials. Some exemptions apply however, such as automotive and industrial electronics, computing and communication infrastructure, medical and military electronics. Second, marketing rewards for “green” laminates also explains the trend to lead-free solder, although usually more expensive. This is one of the reasons why lead-free soldering was implemented very early in Japan. Third, technical requirements such as higher in-use temperature tend to favor higher temperature melting point solders. Conventional lead-based alloys sometimes show limitations due to extensive creep at in-use temperature.

The transition to lead-free soldering should be relatively smooth. It will consist in four major phases, partly overlapping each other. The overall implementation timeline is shown in Figure 1. The implementation forecast proposed in this paper has been developed based on the information collected in different sources, including several reports from Prismark Partner LLC (see the “References” section for details).

- The 1st transition phase relates to Europe and Japan. It already initiated and should be mainly completed by the end of 2007. It involves the consumer electronics, such as audio and video equipments, mobile phones, personal computers, toys. It counted for about 20% of the global electronics industry in 2003.

- The 2nd transition phase involves the same market but in Americas and Asia, that is to say about 22% of the global electronics industry in 2003. It started in 2005 and should grow until the end of 2008.
- The 3rd transition phase relates to the current exemptions, e.g. automotive and industrial electronics, computing and communication infrastructures. These high volume applications cover about 45% of the global electronics industry in 2003. The transition should start in 2006. However lead-free soldering will really be implemented if the exemption rules are revised. Therefore we believe that the transition will still be in progress beyond 2010.
- The 4th phase is for medical and military electronics, which are about 13% of the global electronics industry in 2003. No transition to lead-free is foreseen in the near future.

The lead-free implementation should reach about 60% globally in 2008. Very different situations should be observed in the various geographic areas. The implementation should be completed in Europe and Japan at this date, while it should remain in progress in Americas and in Asia.

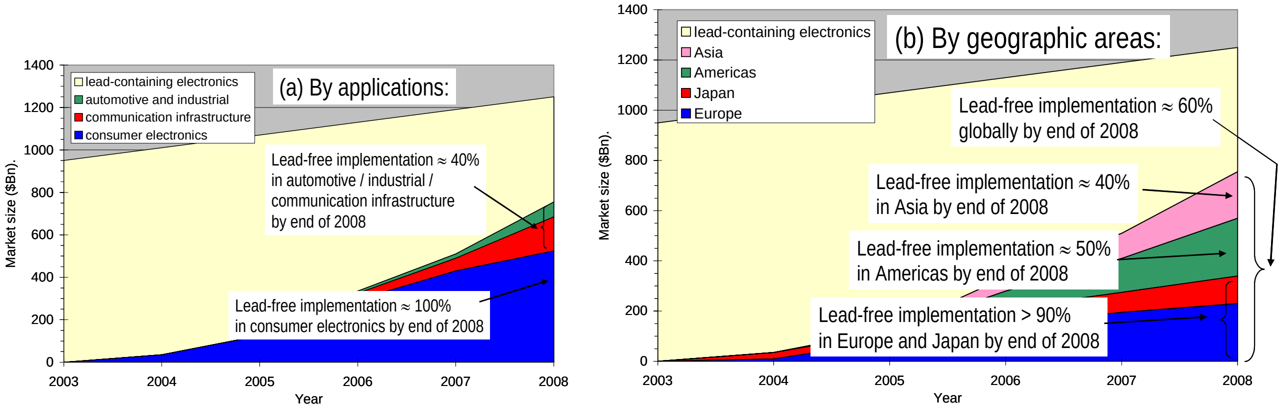


Figure 1 - Implementation to Lead-Free Soldering. (a) Transition by Applications; (b) Transition by Geographic Areas

The soldering temperatures will increase with lead-free solders because most of the lead-free solder alloys melt at higher temperature than that of the eutectic SnPb solder. The change to higher temperature solder alloys will directly affect the temperature profiles for reflow soldering, wave soldering, rework and repair. Typical lead-free reflow profiles will reach peak temperature of 245°C to 265°C, while de-soldering, rework and repair will reach peak temperature above 300°C for a few seconds. Additionally, there is a general trend toward higher complexity of the boards, leading to thicker multilayer structures. Print circuit boards (PCB) will thus be submitted to higher temperature for longer time through multiple reflow cycles. Consequently the laminates used to build lead-free PCB will face new challenges. They will have to fulfill minimum requirements on several key properties:

- Thermal stability, defined by the temperature of decomposition, Td and the time to delamination at 288°C, T-288. It especially controls catastrophic failure during processing (blistering and delamination). It impacts overall in-use reliability as well.
- Coefficient of thermal expansion in the z-direction (perpendicular to the laminate surface), z-CTE. It plays a major role in through-holes reliability (mismatch between the laminate CTE and the copper CTE of the plated through-holes).
- Glass transition temperature, Tg. It improves interconnect reliability (impact on CTE).

In addition to these critical properties, the laminates must maintain sufficient toughness and adhesion to prevent the formation of in-process defects (cracking, delamination) that could lead to reliability issues.

This paper aims to provide correlations between different techniques used to characterize the thermal stability of electrical laminates suitable for lead-free soldering.

- Thermo-Gravimetry Analysis (TGA) was used to measure the degradation temperature (Td) and the time to degradation at a given temperature (D-260, D-288, D-300);
- Thermo-Mechanical Analysis (TMA) was used to measure the time to delamination at a given temperature (T-260, T-288, T-300), the number of temperature cycles before delamination (Nd), and the coefficient of thermal expansion along the z-axis (CTE);
- Differential Scanning Calorimetry (DSC) was used to measure the glass transition temperature (Tg).

The thermal stability data of various epoxy systems will be described and correlated to specific applications needs.

Experimental

Various examples of epoxy systems were chosen within the extensive portfolio of The Dow Chemical Company. They illustrated the diversity of products offering for the specific applications needs. A schematic description of the products positioning is shown in Figure 2. The typical properties of the laminates are described in Table 1. All laminates were flame-retardant, rating UL94 V-0. The FR-4 laminate was chosen as a conventional standard Tg brominated epoxy resin cured with dicyandiamide. The mid Tg LF-150 laminate was made of a brominated epoxy resin cured with a proprietary phenolic hardener. LF-150 stands for "Lead-Free, Tg ≈ 150°C". The LF-150 filled laminate used the LF-150 resin system and 25 phr of silica flour. The LF-170 laminate was the high Tg version of LF-150. LF-170 stands for "Lead-Free, Tg ≈ 170°C". All the LF laminates were designed to fulfill the requirements of the lead-free process. They showed improved thermal stability when compared with FR-4, while maintaining excellent processability and competitive pricing. The HTR-170 system was based on a proprietary technology using a brominated epoxy resin cured with a phenolic hardener. HTR-170 stands for "High Temperature Resistant, Tg ≈ 170°C". It was designed to deliver outstanding thermal performances very suitable for high layer count, good dielectric properties and good toughness. Additional information can be provided by the authors upon request.

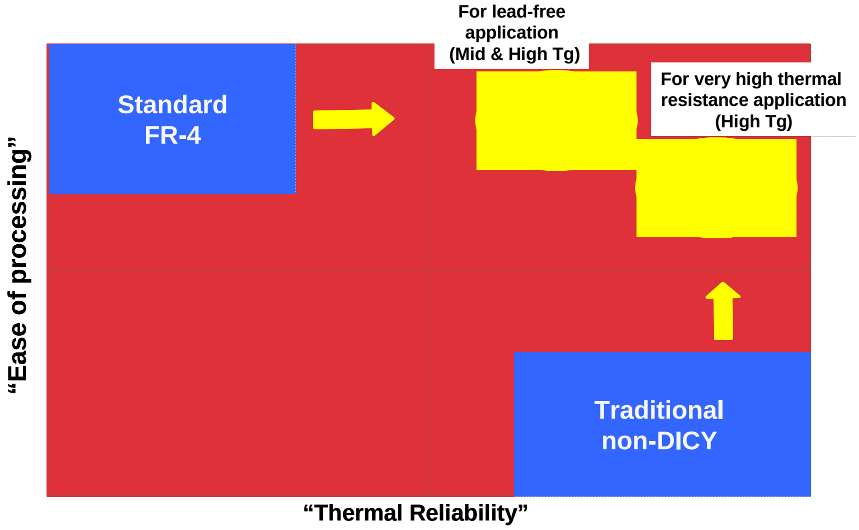


Figure 2 - New Products Positioning

Table 1 - Typical Properties of the Laminates

laminates (1.6 mm)	composition	Tg, °C	Td, °C	T288, min
FR-4	brominated, dicyandimide cure	130 - 140	300 - 310	0 - 1
LF-150	brominated, phenolic cure (incl. proprietary additives)	150 - 160	330 - 340	10 - 15
LF-150 filled	brominated, phenolic cure (incl. proprietary additives), silica filler	150 - 160	330 - 340	10 - 15
LF-170	brominated, phenolic cure (incl. proprietary additives)	170 - 180	330 - 340	10 - 15
HTR-170	brominated, phenolic cure (incl. proprietary additives)	180 - 190	350 - 360	30 +

The glass transition temperature Tg was measured by differential scanning calorimetry using a Mettler Toledo DSC822e, equipped with the sample robot TS0801RO and connected to a personal computer. Runs were performed with a heating ramp of 20°C/min, in nitrogen. Tg was determined by the transition midpoint, following the IPC TM-650-2.4.25C method. The typical experimental error was ± 1°C.

The thermo-mechanical analyzer was a TMA/SDTA840 from Mettler Toledo connected to a personal computer. All measurements were performed in N_2. The time to delamination method was performed according to IPC TM-650-2.4.24.1.

The coefficient of thermal expansion in the z direction (perpendicular to the laminate surface) was done according to IPC TM-650-2.4.24C.

The following procedure was used for the TMA cycling tests:

- Heat to 150°C and equilibrate
- Heat from 150°C to 200°C with a heating rate of 17°C/min
- Heat from 200°C to 260°C with a heating rate of 25°C/min
- Isotherm at 260°C for 0.2 min
- Maximum cooling from 260°C to 200°C with a cooling rate of 15-20°C/min
- Isotherm at 200°C for 3 min

Because of the fast heating rate for the TMA instrument, a short overshoot was observed after the heating to 260°C. Maximum temperature reached up to 277°C for a few seconds before to cool down and equilibrate to 260°C. The overshoot has been taken into account in the modeling described in this paper.

We used a thermo-gravimetric analyzer TGA2950 from TA Instruments, fitted with an auto-sampling device and connected to a personal computer. TGA was usually operated in nitrogen, except if mentioned otherwise (in air). The decomposition temperature Td was measured according to IPC-TM-650-2.3.40, with a heating ramp of 10°C/min (except otherwise mentioned, i.e. 5°C/min). Td was determined at 5% weight loss. The typical experimental error was ± 1°C.

The time to decomposition was determined during isothermal scan at 260°C, 288°C, or 300°C. The temperature was raised to the isothermal measurement temperature with a 10°C/min heating ramp. The time to decomposition was measured at 0.75% weight loss.

The following procedure was used for the TGA cycling tests:

- Heat to 200°C with a heating rate of 10°C/min
- Heat from 200°C to 260°C with a heating rate of 30°C/min
- Isotherm at 260°C for 0.2 min
- Maximum cooling from 260°C to 200°C with a cooling rate of 20°C/min

Results and discussion

Thermo-Mechanical Analysis (TMA)

Thermo-Mechanical Analysis (TMA) was used in the isothermal mode to measure the time to delamination at a given temperature (T-260, T-288, T-300) and the coefficient of thermal expansion along the z-axis (z-CTE). TMA was also used in the dynamic mode to perform cycling tests between 150°C and 260°C. Then we determined the number of temperature cycles before delamination (Nd).

Typical T-288 curves are overlaid in Figure 3. The FR-4 laminate failed after about 1 minute. The sample showed significant delamination after the test. All the LF laminates displayed first delamination after 10 to 15 minutes. The extent of delamination after the test was much less than for the FR-4 laminate. The HTR-170 laminate failed after about 30 minutes. It does not show real delamination after the test but only a few small cracks not visible by eyes.

When the T-260, T-288, and T-300 data were plotted versus the test temperature on a semi-logarithmic scale, straight regression lines were obtained as seen in Figure 4. As a consequence, data suggested an exponential effect of the test temperature on the time to delamination. The correlation between the times to delamination at the different temperature was good for all laminates.

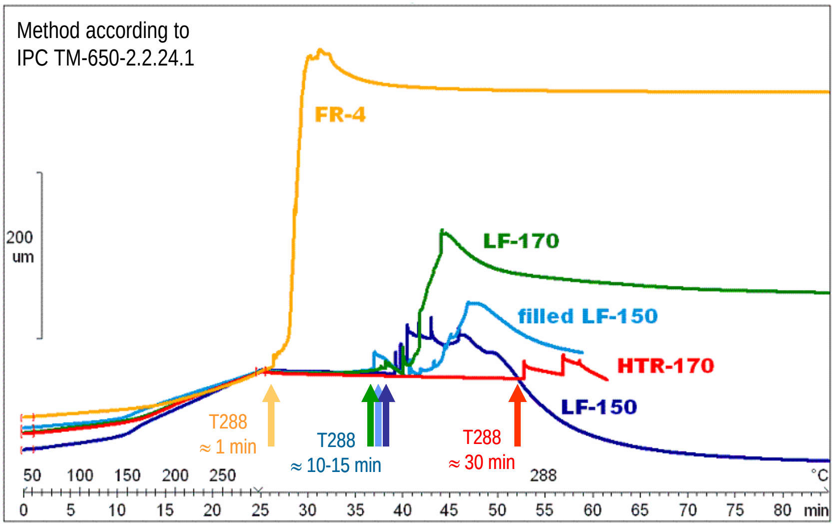


Figure 3 - Typical T-288 Curves for the Different Laminates

The curves we obtained can be considered as the delamination profile for the various laminates. A laminate should not delaminate for a given time/temperature pair if the pair sits below the laminate delamination profile, while it should delaminate above the curve. For instance, a 10 min hold at 275°C should result in delamination for FR-4, while it should not lead to failure for the more thermally stable materials, LF series and HTR-170.

After the isothermal mode, TMA was run in a dynamic mode to perform cycling tests and determine the number of cycles before delamination, Nd. Typical curves are shown in Figure 5. The conventional FR-4 laminate failed after 5 cycles, i.e. Nd = 5. On the contrary, all the laminates with improved thermal stability (LF series and HTR170) do not show evidence of delamination after 15 cycles, i.e. Nd > 15. All the curves of a given laminate perfectly overlaid except for the 1st cycle, probably because of physical network relaxation. Such a relaxation was also observed during the 1st DSC scans.

From these data, it can be concluded that FR-4 laminate should still be the material of choice for simple lead-free applications, with a few reflow cycles only and relatively low laminate thickness. It could fail for more complex lead-free applications, multiple reflows and higher laminate thickness. The thermally resistant products should be the new materials of choice for complex lead-free applications and high layer count.

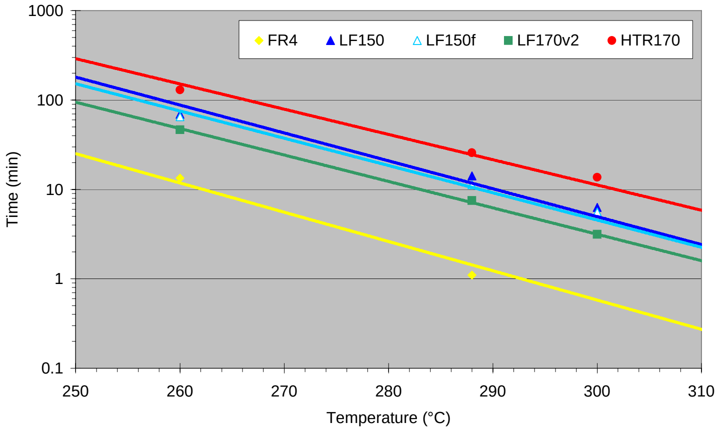


Figure 4 - Time to Delamination Measured by TMA as a Function of the Test Temperature

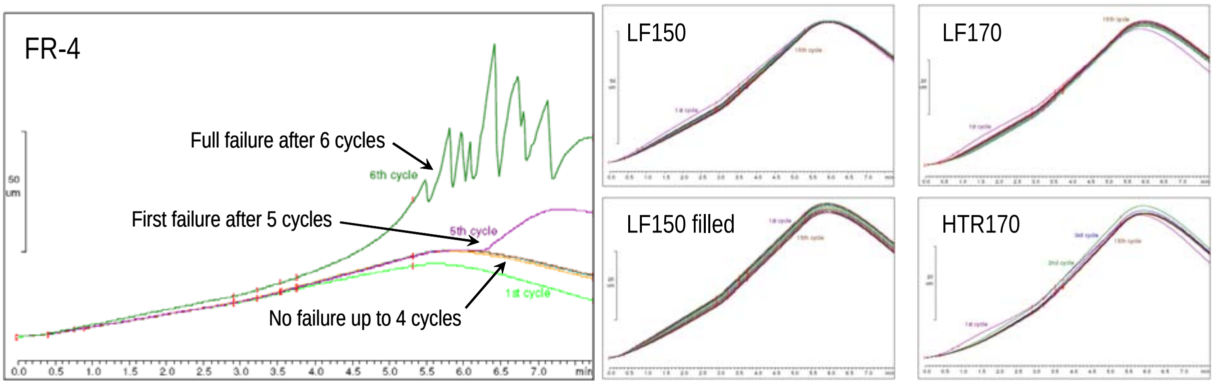


Figure 5 - MA Cycling Tests Results

The envelopes of the cumulative residence time at given temperatures during the TMA cycles and the time to delamination profiles were plotted as a function of temperature on the same chart, displayed in Figure 6. The decomposition profile of FR-4 intersected with the envelope of residence time between the 5th and the 10th cycle. As a consequence, the model predicted a failure between these cycles. This was in excellent agreement with the experimental results. Unlike FR-4, the decomposition profiles of the more thermally stable laminates (LF series and HTR-170) did not intersect with the envelopes of residence time. The model predicted no failure, also in good agreement with the experimental results.

The TMA cycling tests reflected the influence of the coefficient of thermal expansion of the laminates. The effect of Tg and filler on the thermal expansion can be analyzed over a cycle for the LF series, as shown in Figure 7. The TMA cycle started at 150°C, above Tg of LF-150 and below Tg of LF-170. The overall thermal expansion during a cycle was therefore higher for the LF-150 laminate than for the LF-170 laminate. However the difference was relatively small. The impact of filler of the thermal expansion was much more significant, as observed by comparing LF-150 and LF-150 filled. The use of filler was the most efficient way to reduce the coefficient of thermal expansion in the z direction (perpendicular to the laminate surface), while the increase of Tg had only a minor effect.

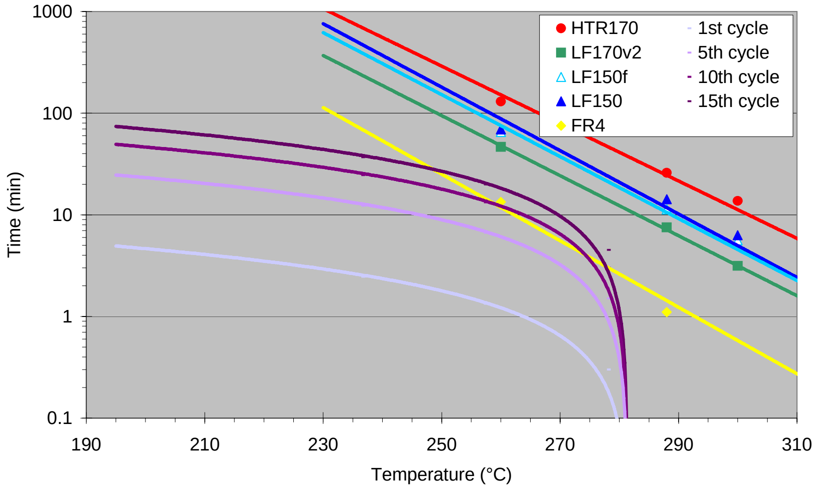


Figure 6 - Prediction of Thermal Resistance during TMA Cycling Test

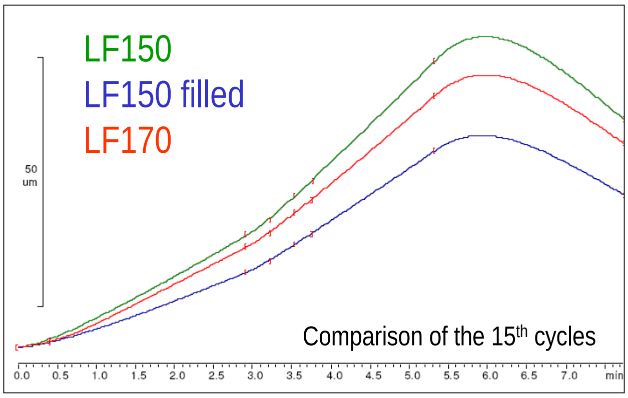
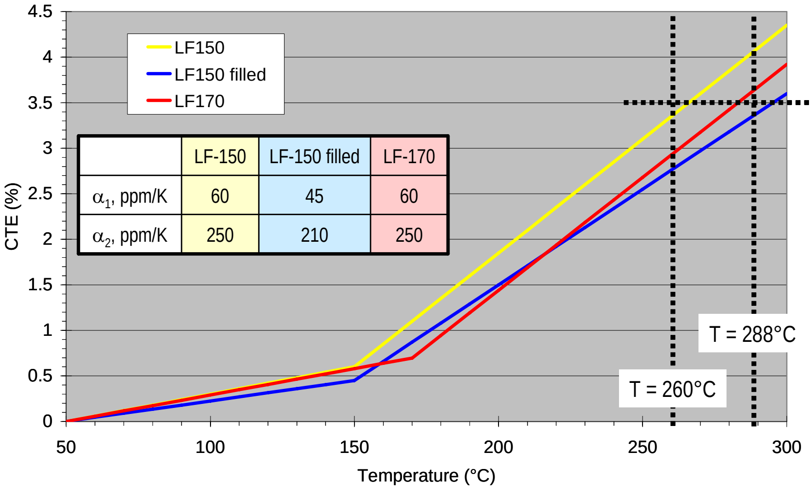


Figure 7 - Effect of Tg and Filler on the Thermal Expansion during a TMA Cycle

The coefficient of thermal expansion in the z direction (perpendicular to the laminate surface) is often described as the overall thermal expansion between 50°C and 260°C, z-CTE [50-260]. With the increase of soldering temperature, the new standard might become z-CTE [50-288], i.e. the overall thermal expansion between 50°C and 288°C. In this case, the coefficient of thermal expansion post-Tg will play an even more significant role. This is schematically described in Figure 8. By considering typical coefficient of thermal expansions for LF-150, z-CTE [50-260] was about 3.4%. The increase of temperature to 288°C led to z-CTE [50-288] equal to 4.1%. The resulting mismatch of thermal expansion between the organic resin of the laminates and the copper barrels will significantly increase. The use of a higher Tg system LF-170 brought down z-CTE [50-288] to 3.7%, assuming similar coefficient of thermal expansions pre- and post-Tg for LF-150 and LF-170. The use of filler in LF-150 allowed the laminate to achieve z-CTE [50-288] equal to 3.4%, similar to z-CTE [50-260] of the unfilled LF-150 laminate. As a consequence, such a filled system should not induce more thermal stress at 288°C than an unfilled system at 260°C. This could be important for the board reliability.



	LF-150	LF-150 filled	LF-170
α_1, ppm/K	60	45	60
α_2, ppm/K	250	210	250

Figure 8 - Schematic Effect of Tg and Filler on the Thermal Expansion Before and After Tg

Thermo-Gravimetry Analysis (TGA)

Thermo-Gravimetry Analysis (TGA) was used in dynamic mode (heating ramp) to measure the degradation temperature (Td) and in static mode (isothermal scan) to determine the time to degradation at a given temperature (D-260, D-288, D-300). TGA was also used in the dynamic mode to perform cycling tests between 200°C and 260°C.

The degradation temperature (Td) of the laminates was measured with 2 heating ramps, respectively 10°C/min and 5°C/min. The results are displayed in Figure 9. The correlation of the Td's obtained with the different heating ramp was excellent. The faster heating ramp led to higher Td's, as expected. It appeared that ΔTd between 10°C/min and 5°C/min was about constant for the laminates tested in this study, equal to 15°C. For these products, Td measured at 5°C/min ≈ Td measured at 10°C/min – 15°C. This relationship seems to be valid for many brominated epoxy systems cured with phenolic hardeners.

All Td's measured in air were lower than the respective Td's measured in nitrogen in the same conditions. The difference was between 2°C and 9°C, depending on the composition. No correlation was performed. The major effect of the TGA gas purge was observed on residual charring at 700°C. The residual weight was much lower in air than in nitrogen because oxygen was necessary to achieve pyrolysis.

TGA was used in the isothermal mode to measure the laminate weight loss as a function of time at 260°C, 288°C, and 300°C. Results are presented in Figure 10. The FR-4 laminate showed early degradation at 260°C. It started to decompose almost immediately at 288°C, and the weight loss was already significant prior to reach 300°C. The laminates from the LF series displayed very limited weight loss during the isotherm at 260°C. They started to decompose at 288°C, and showed early degradation at 300°C. The HTR-170 laminate did not show any significant decomposition at 260°C, limited weight loss at 288°C, and really started to decompose at 300°C.

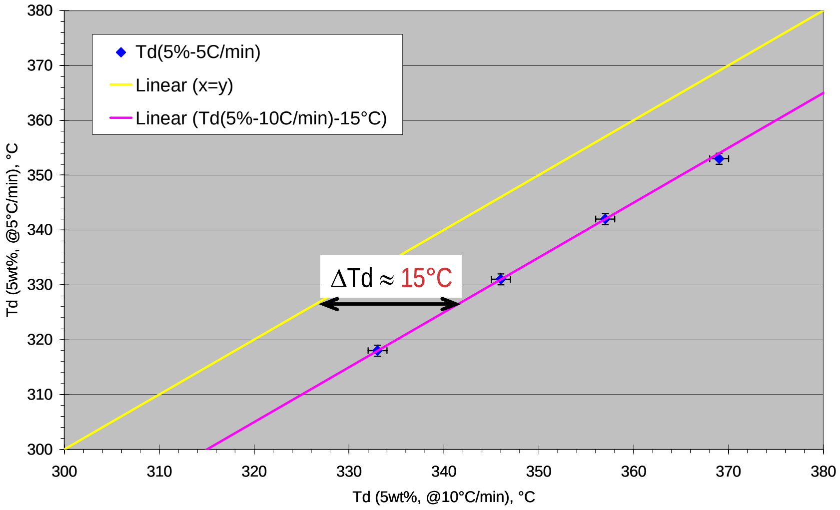


Figure 9 - Effect of the Heating Ramp on Td

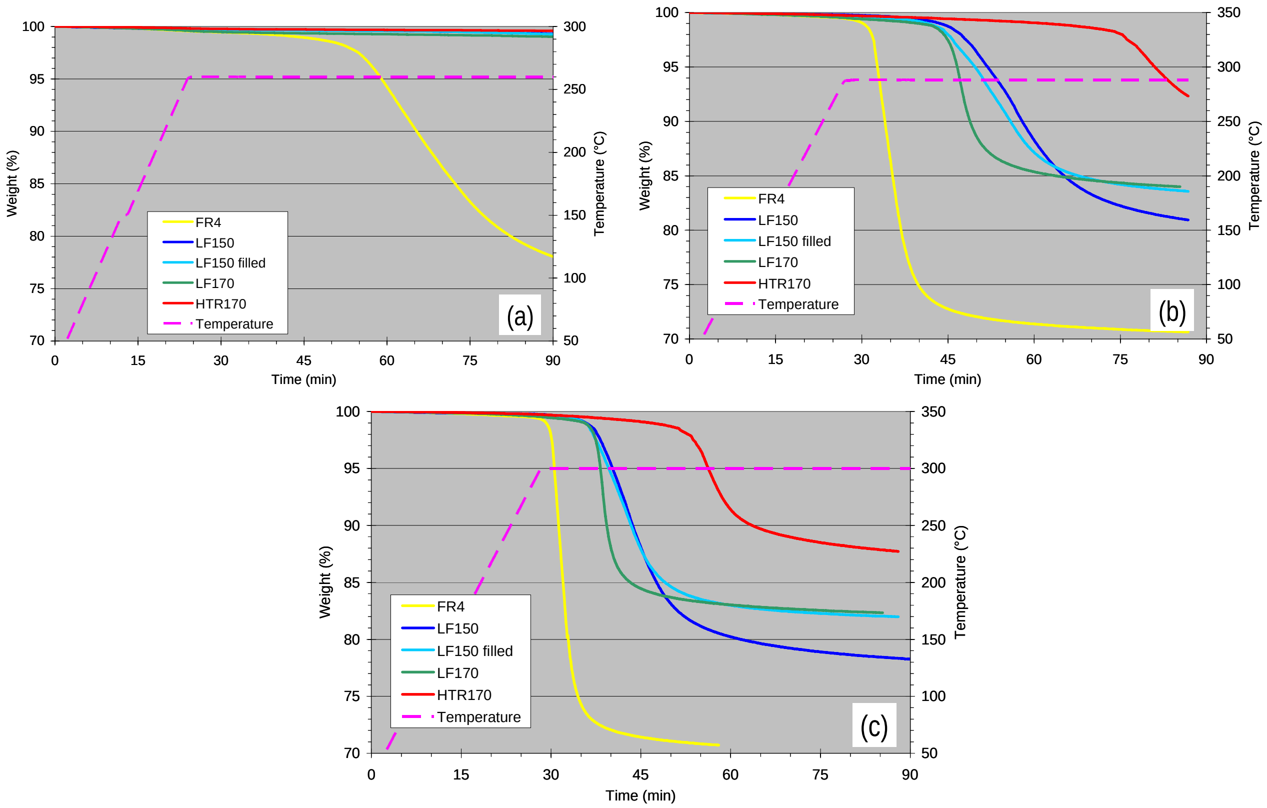


Figure 10 - Laminates Weight Loss vs. Time: (a) at 260°C, (b) at 288°C, (c) at 300°C

The curves of Figure 10 were used to determine the time to decomposition at 260°C, 288°C, and 300°C, respectively D-260, D-288, and D-300. They were measured at 0.75% weight loss, as shown in Figure 11 for D-288. We selected 0.75% weight loss because it gave the best correlation between TMA and TGA results, as described in a later section.

The isothermal scans confirmed that extensive degradation (weight loss) can occur at temperature much below Td measured during a dynamic scan. The decomposition process was clearly time/temperature dependent. During this study, the initial degradation process involved the formation of Br radicals. As already observed by TMA, there was a major change between 260°C and 288°C results.

The residual weight of the different laminates was not significantly changed by the isotherm temperature (in nitrogen) if the hold time was long enough. The residual weight (charring) finally reached a plateau. Apparently the more thermally stable laminates showed more charring, HTR-170 > LF series > FR-4 (in N_2). It was explained by the phenolic hardeners that were better charring promoters than dicyandiamide.

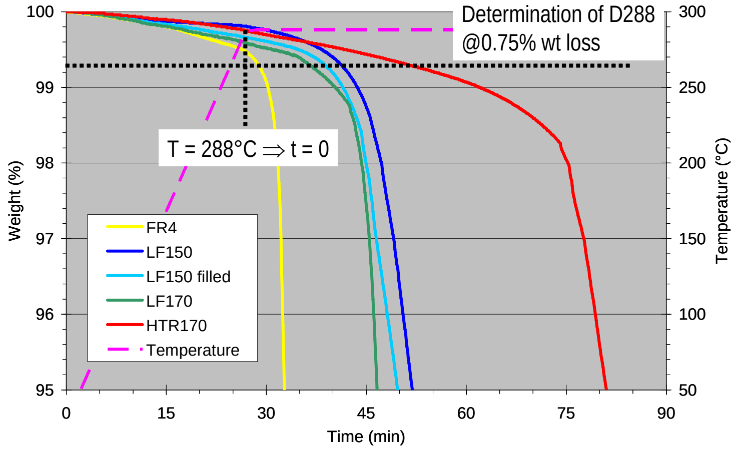


Figure 11 - Determination of D-288

TGA was also used to perform cycling tests between 200°C and 250°C. Residual weight was measured as a function of time for the different laminates. Typical curves are shown in Figure 12.

The cumulative laminates weight loss was plotted as a function of the number of TGA cycles in Figure 13. The FR-4 laminate showed an extensive weight loss during TGA cycles. Moreover an acceleration of the decomposition rate was observed after about 10 cycles, still increasing after 15 cycles. The LF and HTR products showed limited weight loss during TGA cycles. They were still in a "steady-state" after 15 cycles, i.e. the decomposition rate was about constant (no acceleration).

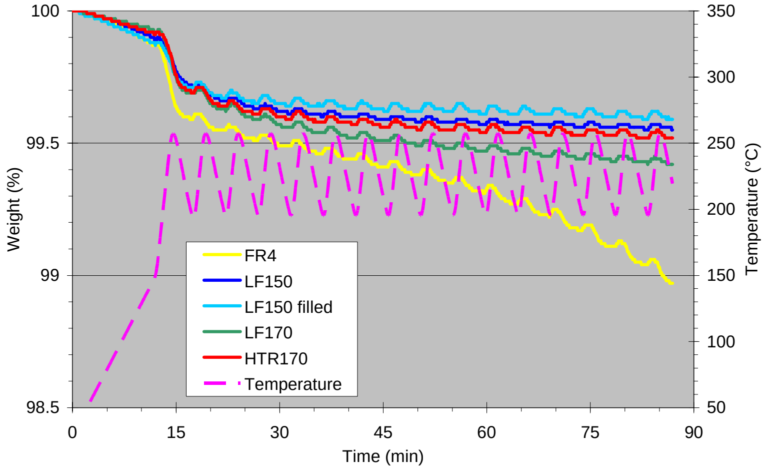


Figure 12 - TGA Cycling Tests Results

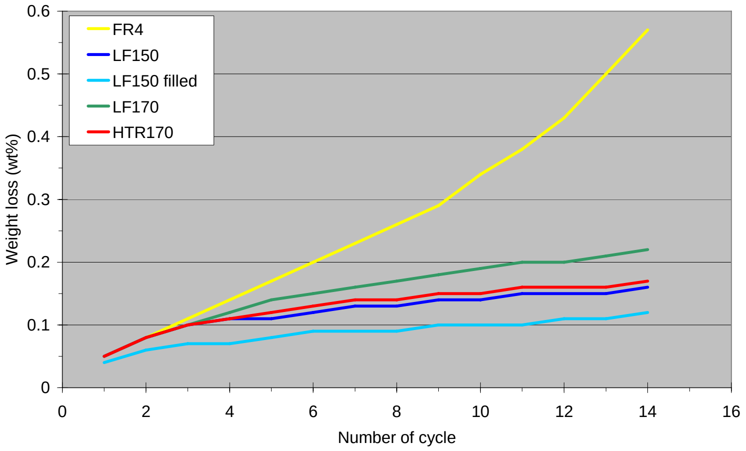


Figure 13 - Cumulative Laminates Weight Loss as a Function of the Number of TGA cycles

Comparison of TMA and TGA results

The time to delamination measured by TMA was plotted as a function of decomposition temperature measured by TGA in Figure 14. There was an excellent correlation between Td and T-260 / T-288 / T-300. The time to delamination at a given temperature, noted T-xyz, was proportional to the exponential of the decomposition temperature

$$T_{XYZ} \propto a \cdot \exp(b \cdot T_d)$$

where a and b are constants for a given TMA temperature.

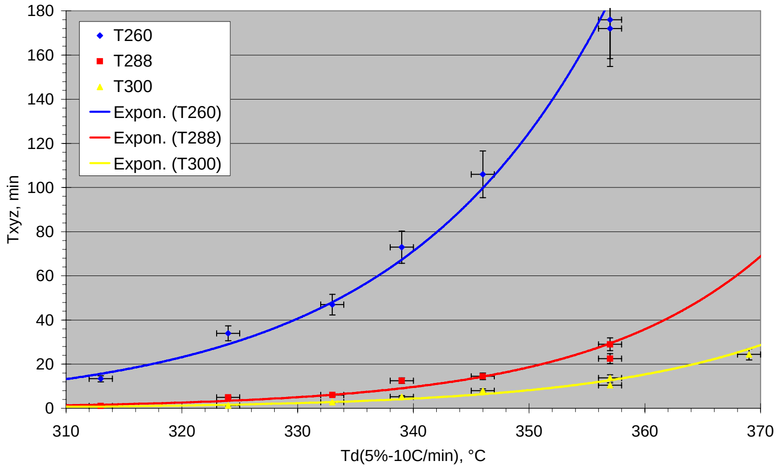


Figure 14 - Time to Delamination Measured by TMA as a Function of Decomposition Temperature Measured by TGA

Based on the best fits obtained in Figure 14, it was possible to model the theoretical time to delamination at a given temperature measured by TMA as a function of the decomposition temperature measured by TGA (10°C/min heating ramp, in N_2). A few examples are given in Table 2.

As explained in a previous section, the time to decomposition at 260°C, 288°C, and 300°C, respectively D-260, D-288, and D-300, was measured at 0.75% weight loss. We selected 0.75% weight loss because it gave the best correlation between TMA and TGA results. It highlighted the fact that the early decomposition species were critical for the overall thermal resistance of the laminates. Because degradation products turned from solid to gas during decomposition, a very small weight fraction can generate a large volume of gas, at the laminate scale. If the decomposition kinetics were too fast for the gaseous compounds to diffuse through the laminate, pressure build-up were produced, leading to delamination. As seen in Figure 15, there was a linear relationship between D-xyz and T-xyz. Most of the results were within the experimental error, i.e. ± 10%. However D-xyz underestimated T-xyz for very high thermal resistant products, probably because of the slower kinetics of decomposition.

Table 2 - Theoretical T-260, T-288, and T300 as a Function of Td (10°C/min, in N_2)

Td, °C	320	330	340	350
T-260, min	23	40	71	> 120
T-288, min	2.6	5	10	19
T-300, min	1.3	2.4	4.4	8

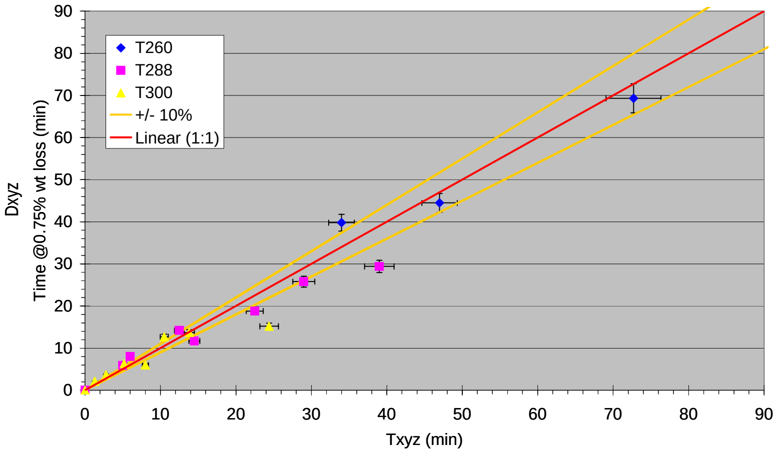


Figure 15 - Time to Decomposition D-xyz Measured by TGA as a Function of Time Delamination T-xyz Measured by TMA

Conclusions

This paper presented experimental results obtained by using various techniques of thermal characterization. It showed that there is an excellent correlation between the time to delamination measured by thermo-mechanical analysis (TMA) at various temperatures, T-260, T-288, and T-300. The results obtained by TMA and by thermo-gravimetry analysis (TGA) also correlated well. The time to delamination by TMA T-xyz was similar to the time to decomposition by TGA D-xyz when D-xyz was measured at 0.75% weight loss. The early decomposition species were critical for the overall thermal resistance of the laminates because they turned from solid to gas during decomposition. A very small weight fraction of degradation products can generate a large volume of gas. If the decomposition kinetics were too fast for the gaseous compounds to diffuse through the laminate, pressure build-up were produced, leading to delamination. However there were also an excellent correlation between T-xyz and Td measured at 5% weight loss (in nitrogen). All the results suggested an exponential effect of the temperature on the decomposition process. Finally the cycling tests performed by TMA or TGA correlated well with the conventional isothermal tests. It was possible to model and predict the maximum number of reflow cycle before delamination.

From these results, we believe that conventional FR-4 laminates will continue to be the most effective base materials for simple applications such as rigid boards with a few reflow cycles. The more thermally resistant laminates will become the materials of choice for more complex applications such as thick boards with multiple reflow cycles. There will be no need to "over-engineer" the laminate materials. Td > 320-330°C should be enough in most cases. Td > 330-340°C might be more suitable in case of demanding process, e.g. high layer count. Tg had no influence on thermal stability and played a minor role in the overall board reliability. The use of filler was the most effective method to lower the coefficient of thermal expansion. It should improve reliability by reducing the expansion mismatch between the organic resin and the copper barrels. In any cases, the improvement of thermal stability must not be done at the expense of the mechanical properties. It will be critical to maintain good adhesion and sufficient toughness to avoid reliability issues.

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