

对PCB制成品性能的评定普遍注重通过和互联可靠性。对热循环或者互联应力的测试也是如此。基底材料有通过或者通不过这些测试的固有能力和，但是PCB制成品的性能永远不会比基底材料更好。这项研究考虑分析测试层压板的新方法。我们发现，在最高温度 235°C 时，层压板材料性能的变化可以量度，而在260°C 时，测试材料之间确定存在分歧。这些变化也许会或者不会对 PCB 性能和可靠性带来真实而且可以量度的损失。

# Thermal Analysis of BASE MATERIALS Through Assembly

Can current analytical techniques predict and characterize differences in laminate performance prior to exposure to thermal excursions during assembly? **by ERIK J. BERGUM**

For PCBs, multiple soldering steps during assembly are essentially standard. A variety of tests evaluate the performance of finished PCBs, and indirectly, the materials and material performance of the substrate materials. These tests, as exemplified by the 6 X 288°C Thermal Shock Test currently in vogue, tend to focus on via and interconnect reliability. These tests are also often combined with lifecycle testing, such as conventional thermal cycling or interconnect stress testing (IST), to gauge in-use reliability performance, again focusing on board reliability.

Substrate materials have an inherent ability to pass or fail

these various tests, but with a high dependence on design and production processes. The maximum ability of a substrate to perform to a certain level can be viewed as the “performance entitlement” of that substrate. A PCB fabricated from a given material can meet the performance entitlement of that material but never exceed it. Our research is primarily an attempt to develop new analytical test methods to determine the performance entitlement of various laminates. Conclusions presented focus primarily on the test methods under investigation and only on the observed differences where the results seem to point to obvious conclusions that are consistent with

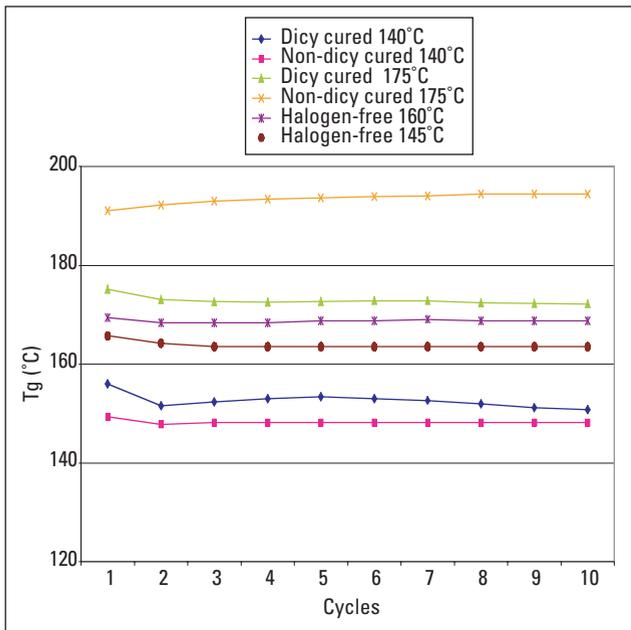


FIGURE 1.

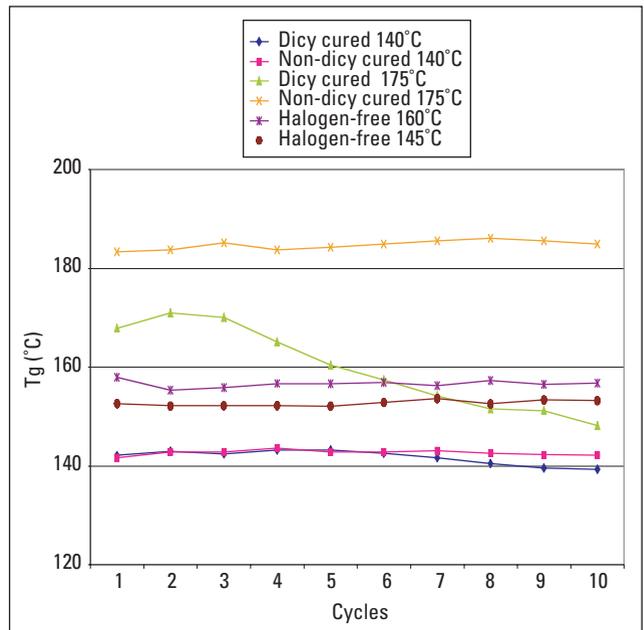


FIGURE 2.

**TABLE 1.** DSC Baseline Data

SAMPLES	T <sub>g</sub> BY DSC		
	PASS 1 (°C)	PASS 2 (°C)	DELTA T <sub>g</sub> (°C)
Dicy cured 140°C	141.46	145.45	3.99
Non-dicy cured 140°C	143.77	146.57	2.80
Dicy cured 175°C	175.43	173.41	-2.02
Non-dicy cured 175°C	183.10	184.91	1.81
Halogen-free 160°C	155.15	155.23	0.08
Halogen-free 145°C	152.62	152.32	0.30

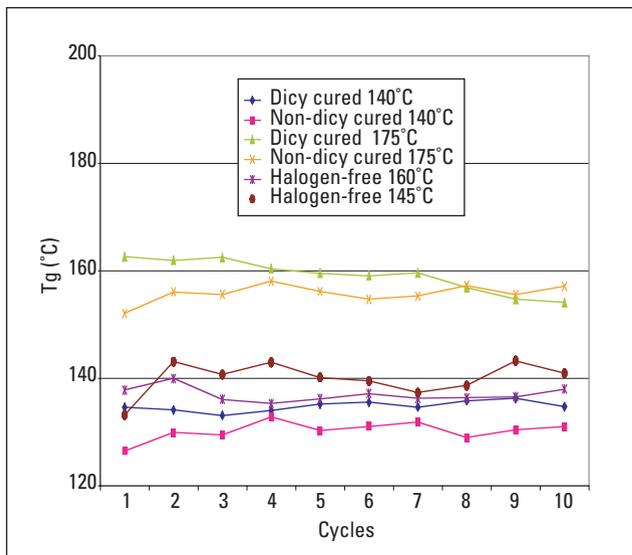
prior art and experience.

**Test Rationale**

The basic premise of this work is to determine if more-or-less standard analytical techniques could be used and adapted to characterize differences in base laminate material performance as impacted by typical thermal excursions experienced during assembly processes. The contention is that the thermal history of a PCB through assembly could be mimicked, to a large extent, using the capabilities of analytical thermal analysis equipment while at the same time measuring specific properties and the changes in those properties with repeated temperature excursions.

In an effort to try to understand the performance entitlement of various materials, assembly cycle simulations were performed by repeatedly thermal-cycling a sample using thermal analysis equipment. The two assembly cycles simulated are typical of cycles used with standard tin/lead solder alloys and with lead-free solder alloy processes. The maximum temperatures reached were 235°C and 260°C respectively, with a dwell time at peak temperature of 10 sec. Heat rise rates during an actual soldering process are very fast compared to the capability of conventional thermal analysis equipment and have a “shock” effect on the PCB. This was not practical to examine or feasible to study using conventional thermal analysis equipment.

Based on this, it was decided to use standard analytical



**FIGURE 3.**

**TABLE 2.** TMA Baseline Data

SAMPLES	T <sub>g</sub> (°C)	TMA RESULTS	
		PRE-T <sub>g</sub> Z (PPM/°C)	POST-T <sub>g</sub> Z (PPM/°C)
Dicy cured 140°C	129.18	42.7	258
Non-dicy cured 140°C	128.40	61.3	256
Dicy cured 175°C	161.57	62.1	275
Non-dicy cured 175°C	168.88	56.9	198
Halogen-free 160°C	139.88	25.4	123
Halogen-free 145°C	139.84	38.8	231

method heat rise rates, which would actually result in longer dwell times above various threshold temperatures and, as such, could be viewed as a worst-case scenario from an exposure time factor if not from a “shock” effect standpoint. Specifically, 10 cycles of differential scanning calorimetry (DSC), thermal mechanical analysis (TMA), thermal gravimetric analysis (TGA) and dynamic mechanical analysis (DMA) using standard, applicable test method conditions were run in sequence on the same sample to simulate multiple soldering operations. The advantage of using standard heat rise conditions lies in the ability to compare test results to other data sets using standard conditions with minimal test-method introduced artifacts. General test method parameters were:

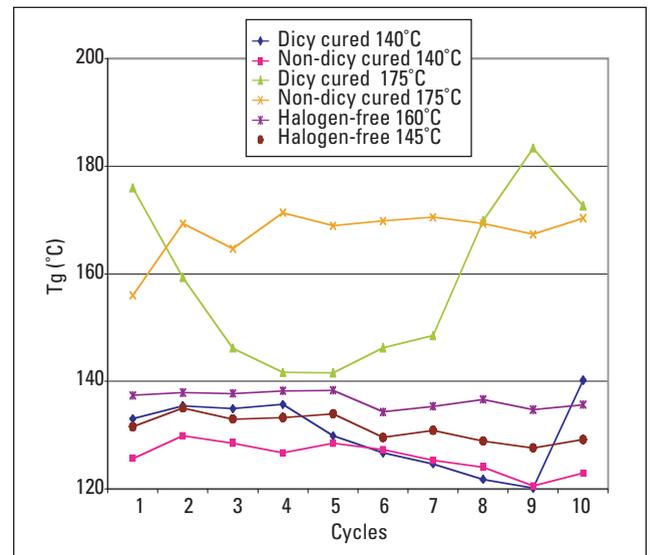
**DSC.** 20°C/min ramp to maximum temperature, isotherm for 10 sec., cool (with copper cladding). Repeat 10 times on the same sample.

**TGA.** 10°C/min ramp to maximum temperature, isotherm for 10 sec., cool (without copper cladding). Repeat 10 times on the same sample.

**TMA.** 10°C/min ramp to maximum temperature, isotherm for 10 sec., cool (with copper cladding). Repeat 10 times on the same sample.

**DMA.** 5°C/min ramp to maximum temperature, isotherm for 10 sec., cool (without copper cladding). Repeat 10 times on the same sample.

The six laminate samples tested were:



**FIGURE 4.**

TABLE 3. TGA Baseline Data

SAMPLES	DECOMPOSITION TEMPERATURE (°C)
Dicy cured 140°C	328.72
Non-dicy cured 140°C	344.55
Dicy cured 175°C	314.46
Non-dicy cured 175°C	344.12
Halogen-free 160°C	346.17
Halogen-free 145°C	354.09

1. A traditional dicyandiamide (dicy) cured, 140°C Tg FR-4 system.
2. A non-dicy cured 140°C Tg FR-4 system.
3. A traditional dicy-cured 175°C Tg high performance FR-4 system.
4. A non-dicy cured 175°C Tg high performance FR-4 system.
5. A halogen-free 160°C Tg high performance FR-4 system.
6. A halogen-free 145°C Tg FR-4 system.

These six materials represent a wide spectrum of FR-4 materials. All testing was done using laminates constructed of eight plies of 7628 glass with a nominal base thickness of approximately 0.059" (1.50 mm). The samples tested were not sufficiently identical that material comparisons based on the baseline data or absolute test values are appropriate other than in general terms. The focus was and should be on the magnitude of change experienced through the 10 cycles. Comparisons of this nature are appropriate based on the intention and methodology of the test.

Data Analysis

DSC results. DSC is the most commonly used thermal analysis method for determining the glass transition temperature (Tg) of a PCB and substrate materials. DSC measures

the rate of heat absorption by a material. Changes in the rate of absorption are used to identify the second order thermodynamic change from a glassy solid to an amorphous solid that is the glass transition. Since other methods such as TMA and DMA measure different property changes associated with the Tg they will, by their very nature, give different results.

Baseline DSC testing of the sample laminates yielded the results shown in TABLE 1. The baseline results are typical of what would be expected for all four materials. Traditionally, a Delta Tg of less than 5°C by DSC is considered a measure of full cure, which all these samples exhibit. However, statistically speaking, a variety of sources, including ASTM, indicate the standard deviation on any single data point based on test variation is between 1.5 and 2°C. Care must be taken to not over-interpret DSC results. With that caveat, FIGURE 1 shows DSC Tg results for all six materials for the DSC 10 assembly cycle simulation at 235°C and FIGURE 2 shows the results at 260°C (Ed.: All figures included in the online version of this article.)

At 235°C, minimal change in the Tg is seen in all samples through the full 10 cycles. At 260°C, the dicy cured 175°C Tg sample shows a reduction in the measured Tg of approximately 12°C after five cycles and 23°C after 10 cycles, suggesting a roughly linear change. From this we can interpret that some aspect of the FR-4 epoxy polymer material, as it affects Tg, is degrading or at least changing when exposed to these conditions on a repeated basis. The other samples again show minimal change throughout the 10 cycles.

TMA results. TMA can also be used to measure Tg but does so by measuring the expansion of the material. A rapid change in the Z-axis expansion rate accompanies the Tg. Most FR-4 and similar thermoset materials show roughly a 500% increase in post-Tg expansion rate vs. the pre-Tg

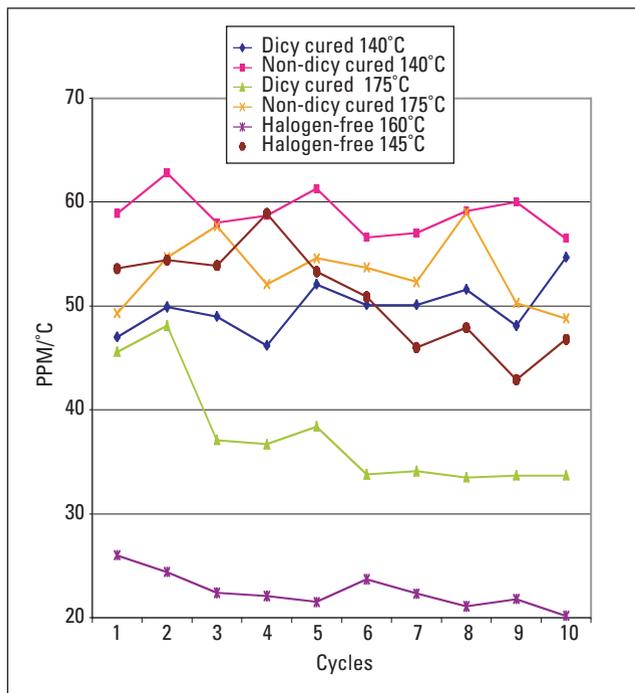


FIGURE 5.

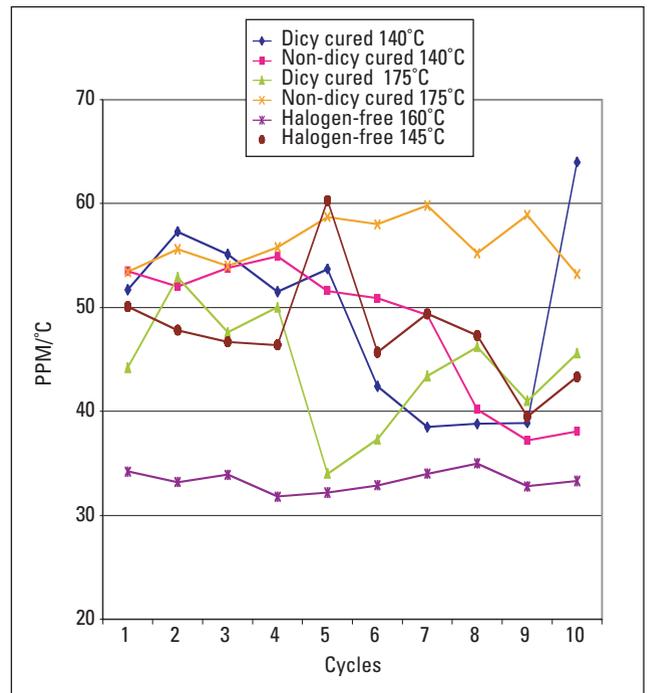


FIGURE 6.

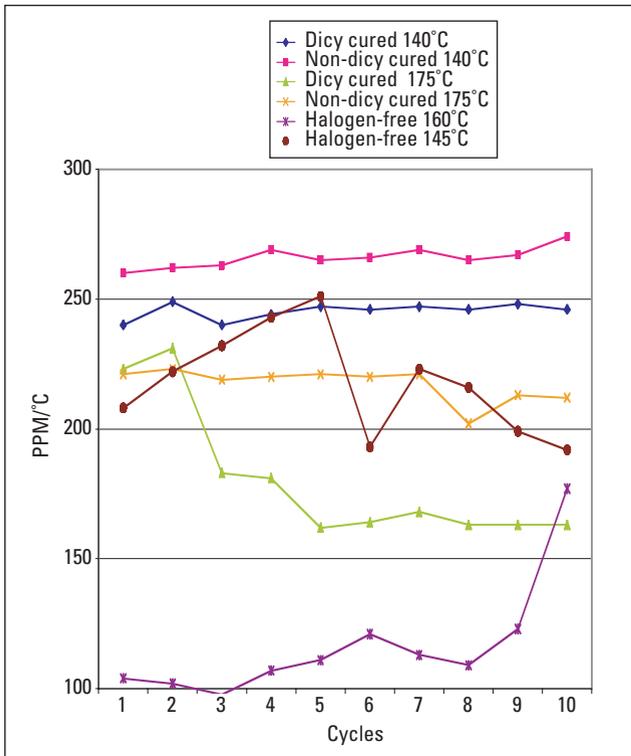


FIGURE 7.

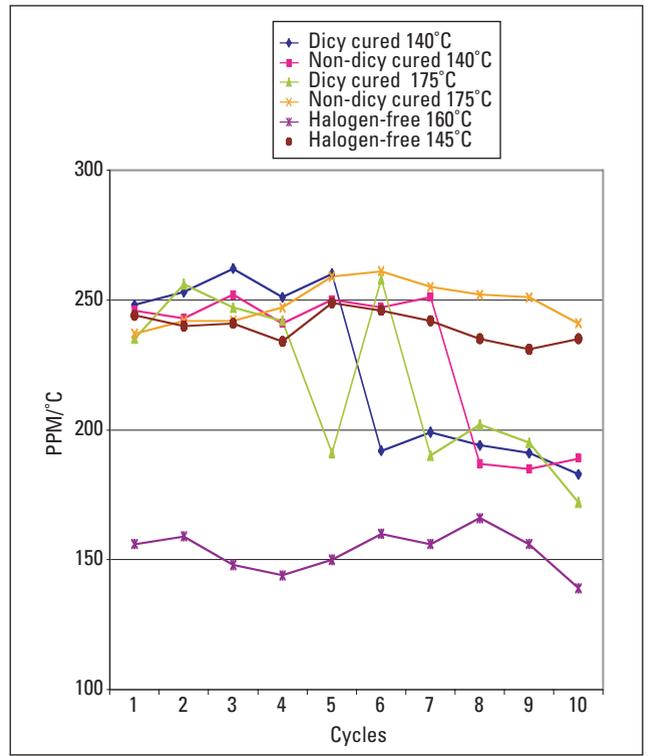


FIGURE 8.

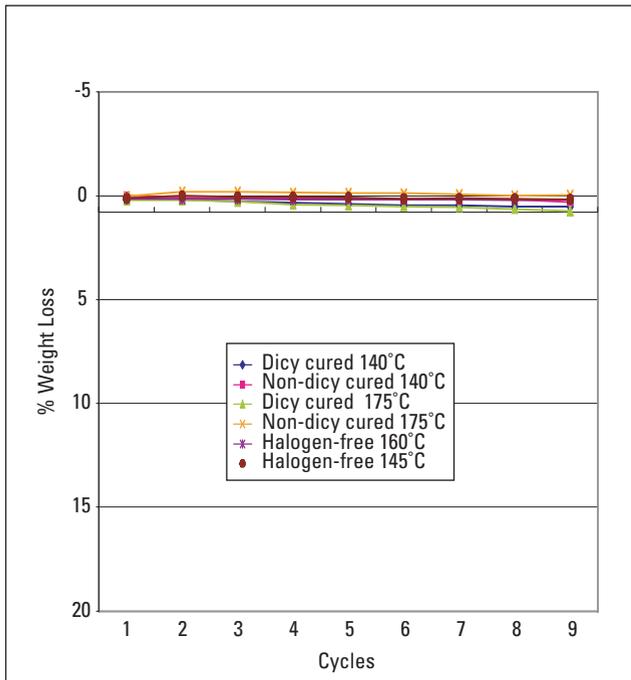


FIGURE 9.

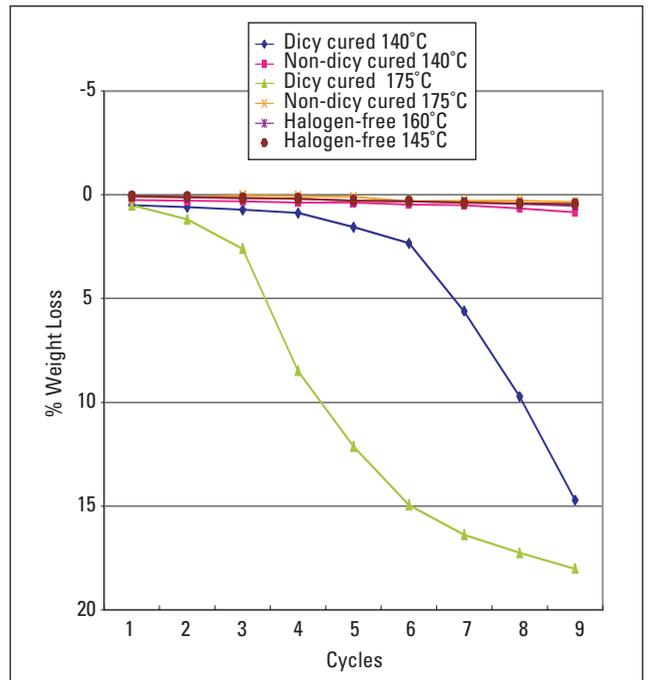


FIGURE 10.

expansion rate. TMA Tg results are typically 5 to 15°C lower than DSC results. Actual expansion rates are highly dependent on resin content of the sample.

TABLE 2 shows the baseline TMA data for the pre-Tg Z-axis expansion rate, post-Tg Z-axis expansion rate and Tg. These baseline TMA results are typical of what would be expected. The only notable points are the post-Tg expansion rate of the non-dicy 175°C Tg sample is and tends to be lower than the other materials tested, and the halogen-free 160°C

Tg sample, which uses an organic “filler” as part of the flame retardant system, also tends to be lower.

FIGURES 3 and 4 illustrate the TMA Tg results for the 10-assembly cycle simulation. As with the DSC results, the TMA Tg results at 235°C show little change through 10 cycles for all materials. This indicates, based on TMA Tg, little or no change in the point at which Z-axis expansion rate changes as related to the Tg. Again, however, at 260°C the dicy 175°C Tg sample shows a distinct reduction in the measured Tg

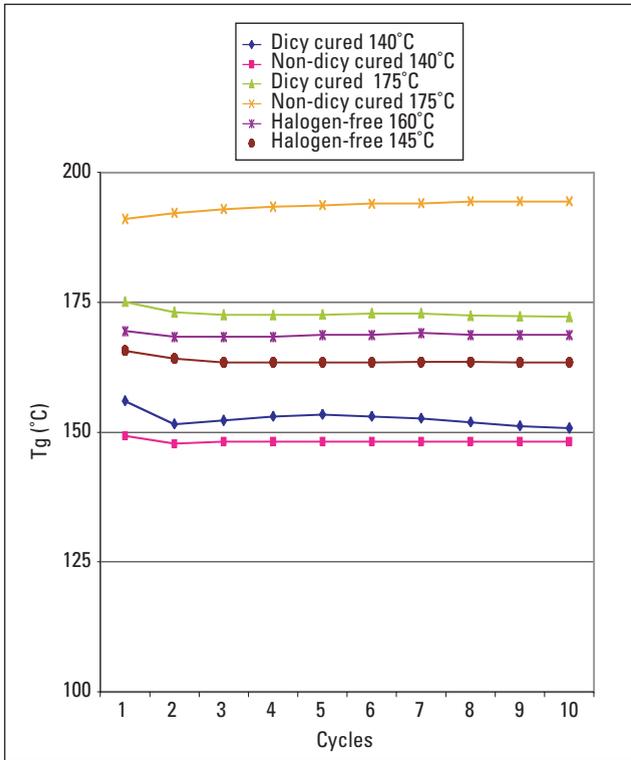


FIGURE 11.

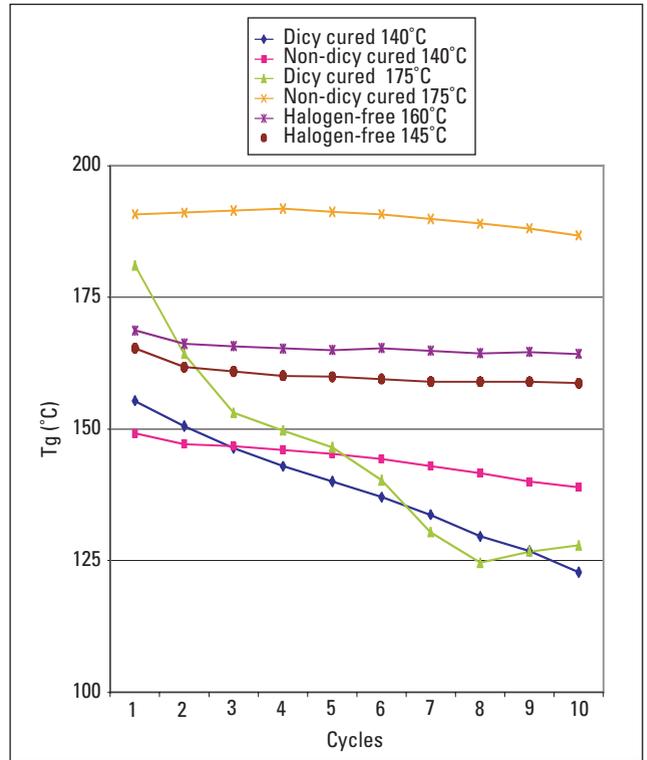


FIGURE 12.

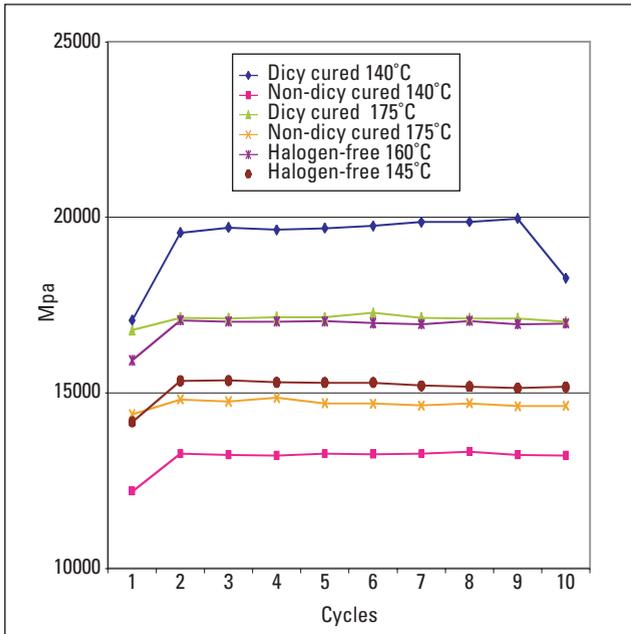


FIGURE 13.

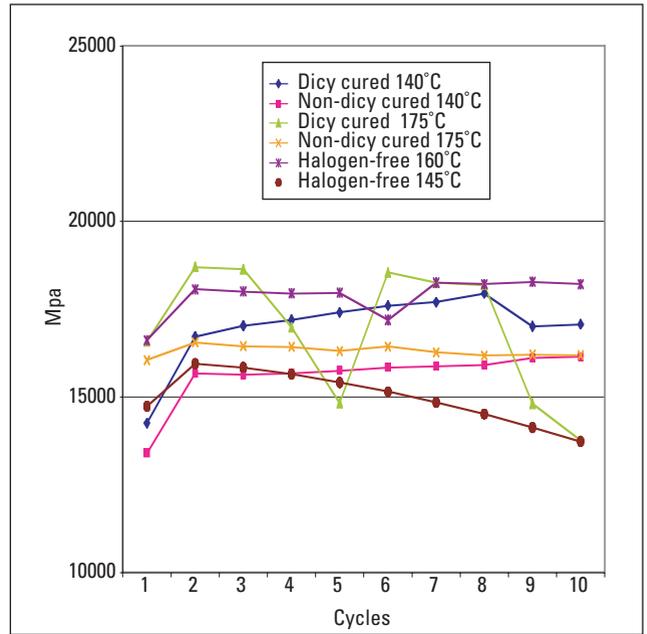


FIGURE 14.

through seven cycles. It is interesting to note, and probably significant, that only this one material shows a distinct reduction in  $T_g$  and of similar magnitude by both TMA and DSC. The rebound in  $T_g$  in cycles eight to 10 is not easily explained.

The pre- $T_g$  expansion rates were also measured as a part of the TMA testing as shown in **FIGURES 5 and 6**. Expansion rates at 235°C, again, prove to be relatively flat with minimal change for all materials tested. At 260°C we see considerable noise in the data with a general decrease for all materials

except the non-dicy 175°C materials. These changes may be large enough to be significant and result from stress relaxation or similar phenomena, which may be associated with a fundamental change or degradation of the polymer.

Post- $T_g$  expansion rates are shown in **FIGURES 7 and 8**. The post- $T_g$  expansion rates show similar results to the pre- $T_g$  results and are observed for the same potential reasons. The changes observed are most notable with the two low- $T_g$  materials.

**TGA results.** TGA is most commonly used to measure the

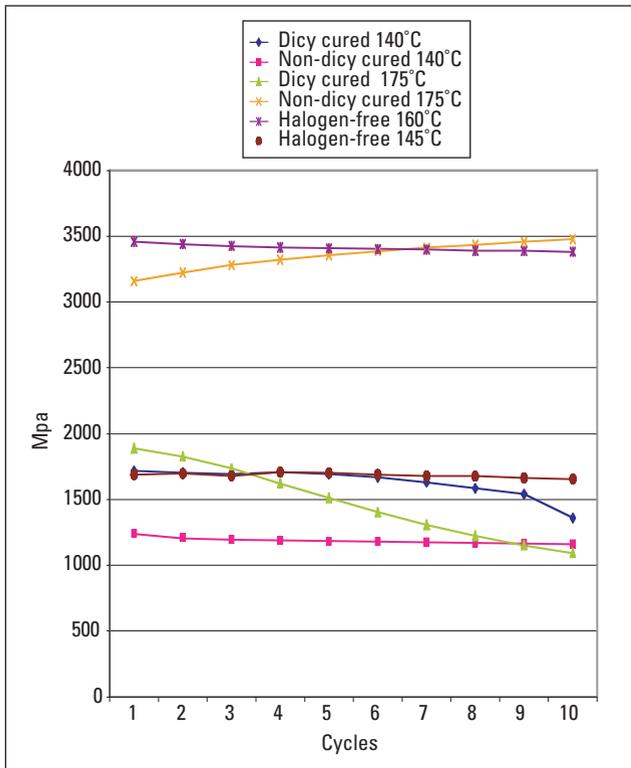


FIGURE 15.

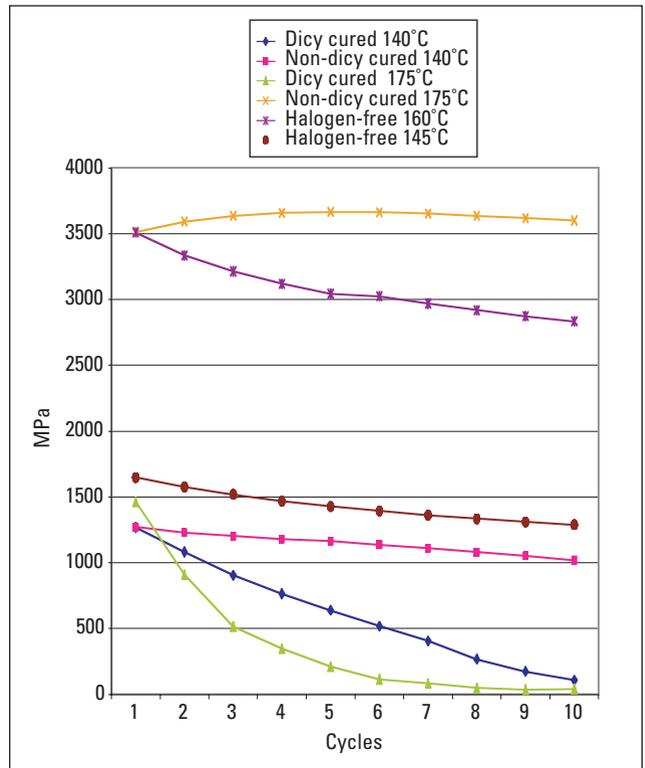


FIGURE 16.

degradation temperature of a material. TGA measures the change in the weight of a sample versus temperature. The degradation temperature in the PCB industry is historically defined as the temperature at which a 5% weight loss occurs. Degradation of a material occurs over a wide temperature range and is the result of irreversible breakage of chemical bonds within the polymer structure. The higher the degradation temperature of a material the more thermally stable the material.

TABLE 3 shows baseline data for TGA decomposition temperature. These baseline values are all typical of the materials tested. The two non-dicy cured materials have inherently higher degradation temperatures due to the use of a non-dicy curing agent, which is more thermally stable. Likewise, the two halogen-free materials use more thermally stable base resins than the classic brominated systems resulting in higher decomposition temperatures.

FIGURES 9 and 10 show the weight change percentage through the 10 TGA cycles. FIGURE 9 shows minimal change for any of the materials through the ten cycles at 235°C. FIGURE 10 shows a very different picture. Both of the dicy-cured samples show very large weight changes throughout the 10 cycles indicating what can be presumed as extensive degradation of the resin.

**DMA results.** DMA measures the flexural properties of a material. The Tg is accompanied by a rapid reduction of the flexural strength or, more properly, storage modulus. DMA typically gives Tg values 5 to 15°C higher than DSC.

TABLE 4 shows the baseline DMA data. Again, these results are typical of what would be expected for DMA for these materials.

FIGURES 11 and 12 depict the DMA Tg results. As with the previous Tg results from the other test methods, minimal

change is observed at 235°C. Again, as with the DSC and TMA results at 260°C, a significant decline is seen for the two dicy-cured materials.

The actual flexural performance of the materials can also be measured by DMA. FIGURES 13 and 14 show this as measured at 50°C and FIGURES 15 and 16 at the peak temperatures of 235°C and 260°C respectively. Little change is noted over the 10 scans at 50°C at either peak temperature that seems to lead to useful or meaningful differentiation between the materials. However, the storage modulus at the peak temperature, and in particular at 260°C, shows significant changes in performance. As one would expect, based on the different material Tgs, peak storage modulus values vary widely. FIGURES 17 and 18 show this peak temperature storage modulus as a cumulative percentage change throughout the 10 cycles. At 235°C some change, on a percent basis, in the performance of the materials is resolvable, but at 260°C the changes in performance become very apparent.

## Conclusions

The assembly simulation and results described show some evidence that at a peak temperature of 235°C laminate material performance changes are measurable using these techniques. The 260°C data definitely show that differences between the materials tested are distinguishable by these techniques. These changes may or may not translate into real and measurable PCB performance and reliability loss.

This study seems to establish proof of concept as to the ability of these techniques to differentiate between resin system performance entitlements. However, considerable work remains in establishing specific and industry-accepted test methods, let alone data-based critical performance levels or cri-

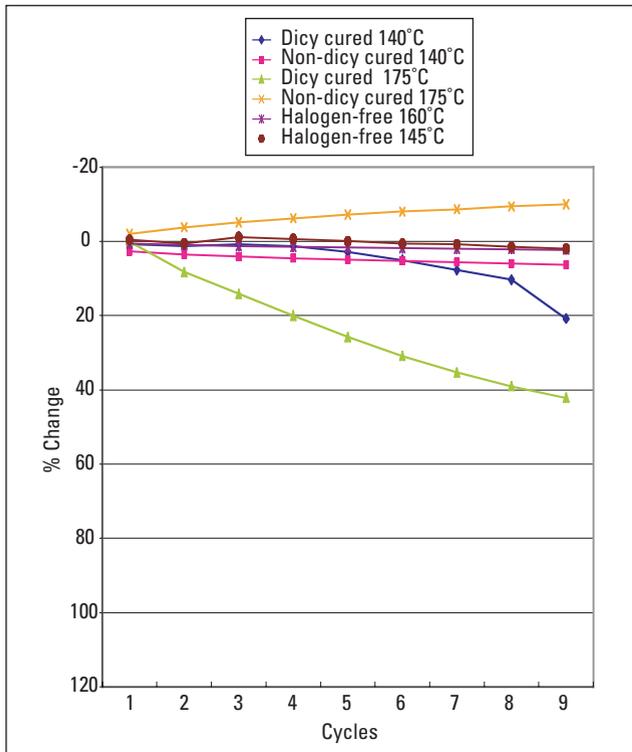


FIGURE 17.

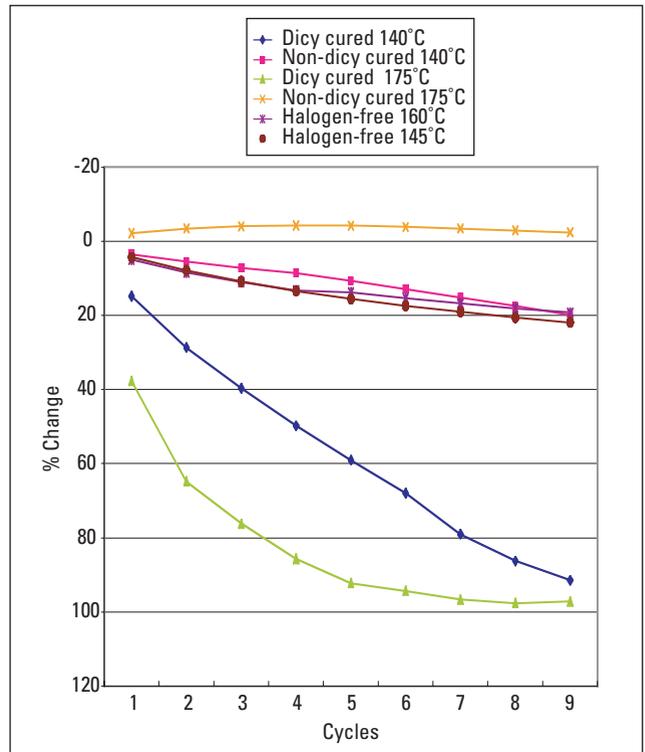


FIGURE 18.

teria. The logical extension is to correlate these changes to PCB performance and laminate material performance entitlement.

Some other conclusions based on the data that seem appropriate are:

1. Since the different thermal analysis techniques all measure very different properties of the materials, great differences exist in observed changes and the magnitude of changes.
2. Again, based on the different properties being measured, it seems unlikely that a single test is capable of providing all relevant information about a material's performance entitlement.
3. All the techniques examined were able to in some way measure differences between the performances of at least some of the samples tested.
4. Testing performed at 260°C showed much greater resolution in the changes observed between the various samples tested.
5. While the changes, or lack thereof are quantitative in nature comparisons can only be made on a qualitative basis lacking baseline or "acceptance" criteria.
6. All the techniques examined seem to have some utility for comparing property changes through the 10-cycle simulation.
7. Materials do have fundamentally different performance property entitlements based on this test rationale.
8. These tests specifically measure changes in material properties, but it is reasonable to assume that these changes are indicative of material performance changes affecting PCB performance.

Additional observations and conclusions that can be made are:

1. Tg alone is a poor predictor of the ability of a material to maintain its original performance properties through the 10 cycles simulated.

2. No single "as is" test or criteria is likely sufficient to determine the performance of different resins.
3. Peak temperature storage modulus by DMA seems to be the best single technique for resolving performance differences between the materials.

### Recommendations and Future Work

Evaluation of actual production PCBs in a similar test regime seems to be warranted based on these data and conclusions. The contribution of the various chemical, mechanical and thermal process steps involved in the production of circuit boards has been well documented by many sources and is generally acknowledged. This baseline should give a very good point from which to measure those overall process influences.

With the move toward lead-free higher temperature assembly processes, additional work of this nature and on PCBs at higher temperatures seems warranted and is planned. **PCD&M**

*Ed: This article is adapted from a presentation at IPC Expo, March 2003, and is used with permission of the authors.*

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