Reengineered FR-4 Base Materials for Improved Multilayer PCB Performance

by Ed Kelley, Polyclad Laminates/Cookson Electronics

Introduction: The Continuing Evolution of FR-4 Base Materials

In overly simple terms, base materials for printed circuits typically consist of three apparently simple components, the copper foil, the resin system, and the reinforcement. However, a closer examination of the materials currently available in each of these categories, as well as a review of the evolution of these materials over the years reveals just how complex base materials can be. Given the many and ever-increasing requirements placed on base materials by printed circuit fabricators, printed circuit assemblers, and OEMs, it should come as no surprise that the components and base laminates have become more complex and varied. The challenge to the supplier of base materials is to provide value to the supply chain by balancing the multiple requirements of each level with products that can be produced cost effectively.

The history of FR-4 base materials is really quite amazing. How many times have people speculated that the life of FR-4 materials was coming to an end, only to see improved versions introduced? Each time the requirements have increased, the suppliers of materials have responded with enhancements that met the new demands. In recent years, the most obvious trend has been the increased use of higher-T_g materials. In fact, many discussions on T_g seem to equate higher-T_g's with higher performance or reliability. One of the primary purposes of this article is to describe other properties that must be considered for the next generation of FR-4 products, and to present data on materials that have been developed to meet the new demands.

Industry Trends Affecting Base Material Requirements

Several trends are underway which will force reengineered materials to be introduced and adopted. These include:

- Multilayer PCB Design Trends
- Environmental Regulations
- Electrical Performance Requirements

One of the most fundamental trends in PCB design is towards increased circuit density. Basically, there are three methods to achieve increased circuit density. First, you can put more circuitry in a given area by decreasing line widths and spacings. Second, you can increase layer counts. Last, you can reduce PTH diameters and pad sizes. Putting more circuitry in a given area can result in higher operating temperatures. Increasing layer counts implies either greater overall thickness, or thinner dielectric spacings with the same thickness. If the PCB is made thicker, thermal cycles will place increased stress on the PTH as a result of the higher total thermal expansion. If thinner dielectric spacings are chosen, this will typically involve the use of base laminates and prepregs with higher resin contents. Higher resin contents will also result in increased thermal expansion and stress on the PTH. Reducing PTH diameters will further increase the plated via aspect ratio. To insure PTH reliability, base materials with reduced levels of thermal expansion and greater thermal stability are required.

In addition, as component densities increase, the need to place plated vias closer together in the PCB also increases, resulting in increased concerns that conductive filaments could form and bridge the distance from hole wall to hole wall. This conductive anodic filament (CAF) formation is the subject of much research and new base materials must be more resistant to this phenomenon.

Environmental regulations are also placing additional requirements upon base materials. The European Reduction of Hazardous Waste (RoHS) and Waste from Electrical and Electronic Equipment (WEEE) directives will significantly affect the requirements placed upon base materials. Among other elements, RoHS restricts the use of lead (Pb). Tin/lead (Sn/Pb) alloys have been used for many, many years in the assembly of printed circuits. Eutectic Sn/Pb has a melting point of 183°C and temperatures during assembly commonly reach 230°C. The primary alternatives to Sn/Pb are tin/silver/copper (Sn/Ag/Cu or "SAC") alloys. These alloys have melting points near 217°C with typical peak assembly temperatures

reaching 255-260°C. This increase in assembly temperature coupled with the possibility of multiple exposures to these temperatures require base materials with improved thermal stability. Recent technical papers have presented important data on the effect of Pb-free assembly on base materials[i],[ii].

The RoHS directive also restricts the use of specific halogenated flame retardants. These include polybrominated biphenyl (PBB) and polybrominated diphenyl ether (PBDE). RoHS does *not* restrict the use of tetrabromobisphenol A (TBBPA), which is the most commonly used flame retardant used in PCB base materials. However, some OEMs are considering the use of completely halogen-free materials due to concerns about the improper incineration of materials containing TBBPA.

High speed, broadband and wireless applications are driving the need for base materials with improved electrical performance. "Improved" can mean stable, predictable and/or lower values for the dielectric constant (D_k) and dissipation factor (D_f). Materials that offer improved thermal performance with essentially the same values of D_k and D_f as currently available FR-4 materials will be in very high demand. In addition, materials with improved thermal performance and lower values of D_k and D_f are expected to experience growing demand.

Important Base Material Properties

While there are many important properties to consider, there are a few that deserve special attention in light of current trends and the resulting need for improved thermal performance. These include:

- The glass transition temperature (T_g),
- Coefficients of thermal expansion (CTEs),
- Decomposition temperature (T_d),

The most commonly referenced base material property used to classify materials is the glass transition temperature, T_g[iii]. The T_g of a resin system is the temperature at which the material transforms from a relatively rigid or "glassy" state, to a more deformable or softened state. T_g is important to understand since the properties of base materials are different above the T_g versus below the T_g.

All materials undergo changes in physical dimensions in response to changes in temperature. The rate at which base materials expand is much lower below the T_g than above. Thermomechanical analysis (TMA) is a procedure used to measure dimensional changes versus temperature. Extrapolating the linear portions of the curve (see Figure 1) to the point at which they intersect provides a measure of the T_g. The slopes of the linear portions of the curve above and below the T_g represent the respective rates of thermal expansion, or as they are typically called, the coefficients of thermal expansion (CTE's). CTE values are important since they influence the reliability of the finished circuit. Other things being equal, less thermal expansion will result in greater circuit reliability as less stress is applied to plated holes. While the T_g is typically described as being a very precise temperature, this is somewhat misleading, since the physical properties of the material can begin to change as the T_g is approached. This explains the curved line in Figure 1.

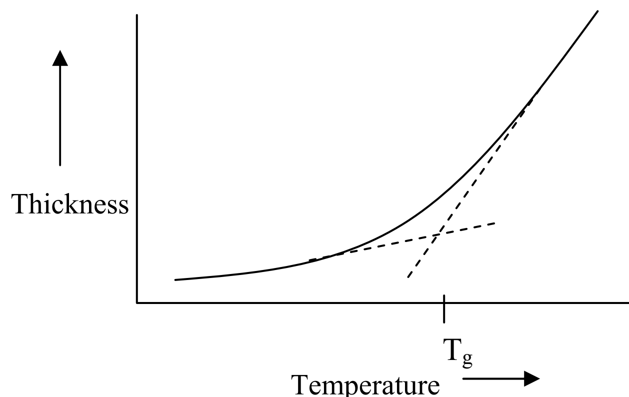

Figure 1 – Measuring T_g by Thermomechanical Analysis (TMA)

Besides TMA, two other thermal analysis techniques are also commonly used for measuring T_g. These are Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA). DSC measures heat flow versus temperature rather than the dimensional changes measured by TMA. The heat absorbed or given off will also change as the temperature increases through the T_g of the resin system. T_g measured by DSC is often somewhat higher than measurements by TMA. DMA measures the modulus of the material versus changes in temperature.

Implicit in many discussions of T_g is the assumption that higher values of T_g are always better. This is not always the case. While it is certainly true that higher values of T_g will delay the onset of high rates of thermal expansion for a given resin system, total expansion can differ from material to material. A material with a lower T_g *could* exhibit less total expansion than a material with a higher T_g, due to differing resin CTE values or, for example, by incorporating fillers into the resin system that lower the CTE of the composite material. This is illustrated in Figure 2. Material C exhibits a higher T_g than material A, but material C exhibits more total thermal expansion because its CTE value above T_g is much higher. On the other hand, with the same CTE values above and below the T_g, the higher- T_g material B exhibits less total thermal expansion than material A. Finally, although the Tg values are the same, material B exhibits less total expansion than material C due to a lower CTE value above T_g.

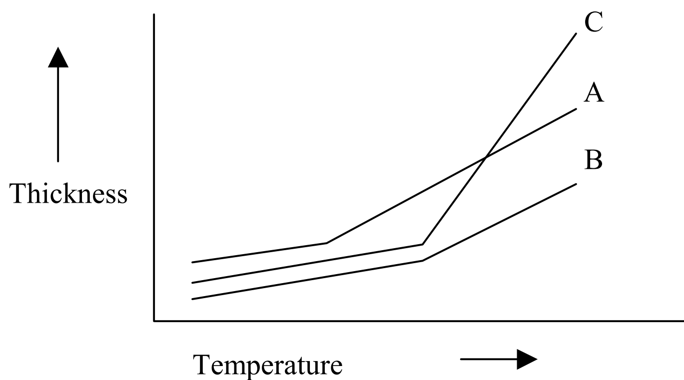

Figure 2 – Impact of T_g and CTE Values on Total Expansion

Figure 3 compares three different 175°C Tg materials. While each material has the same value for T_g, they exhibit differences in overall Z-axis expansion as the result of differences in the respective rates of thermal expansion. In this example, the overall differences are primarily related to the differences in post- T_g rates of thermal expansion. Other things being equal, lower levels of thermal expansion will improve PTH reliability.

However, other things are not always equal! But before we discuss the other properties that must be considered, let me summarize the key points regarding T_g and thermal expansion:

- One of the primary benefits of higher- T_g materials is the general decrease in Z-axis expansion that results. By delaying the onset of the more rapid post- T_g thermal expansion rate, higher- T_g materials generally exhibit less total thermal expansion than lower-Tg materials, resulting in less stress on PTH's.
- However, while admittedly not common, it is possible for higher- T_g materials to exhibit more overall thermal expansion than lower- T_g materials as a result of differences in rates, or coefficients of thermal expansion (CTEs). Therefore, CTE values should also be evaluated when selecting materials.
- Materials with the same T_g value can exhibit different CTEs, and therefore result in different levels of stress applied to PTHs during thermal cycles. Materials such as product C in Figure 3 combine the advantages of both higher- T_g *and* lower CTEs.

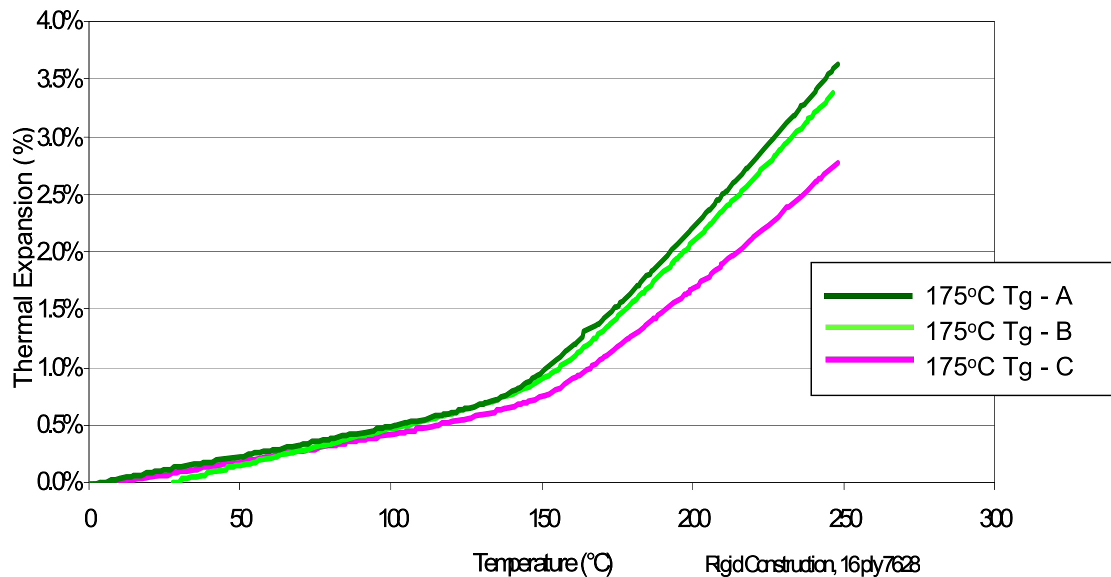

Figure 3 – Z-Axis Expansion for Three Materials with the Same T_g

As the temperatures to which printed circuits are exposed to increase, as in Pb-free assembly processes, the decomposition temperature (T_d) of the material becomes a much more critical property to understand[iv]. The decomposition temperature is a measure of actual chemical and physical degradation of the resin system. This test uses thermogravimetric analysis (TGA), which measures the mass of a sample versus temperature. The decomposition temperature is reported as the temperature at which 5% of the mass of the sample is lost to decomposition. Figure 4 provides an example of two FR-4 materials with the same glass transition temperature, but different decomposition temperatures. Experience is showing that the decomposition temperature is a critical property, and appears to be at least as important, if not more important than the glass transition temperature when planning for Pb-free assembly conversion.

While the definition of the decomposition temperature uses a weight loss value of 5%, it is very important to understand the point at which 2-3% weight loss occurs, or where the onset of decomposition begins. In examining Figure 4, you can see that traditional Sn/Pb assembly processes can reach peak temperatures of 210°C to 245°C, with 230°C a very common value. In this range, neither material exhibits significant levels of decomposition. However, if you examine the temperature range where Pb-free assembly processes are operating, you can see that the traditional material is exhibiting a 2-3% weight loss. Severe levels of degradation can result from multiple exposures to these temperatures.

In order to evaluate the effect of multiple thermal exposures to standard Sn/Pb assembly temperatures in comparison to SAC Pb-free assembly temperatures, the three materials from Figure 3 were run through thermal cycles in a thermogravimetric analysis (TGA) unit[v]. These materials all have Tg values of 175°C, but product A has a decomposition temperature of 310°C while products B and C have decomposition temperatures of 350°C. Figures 5 and 6 show the cumulative weight loss results obtained when cycling these materials to 235°C and 260°C, respectively.

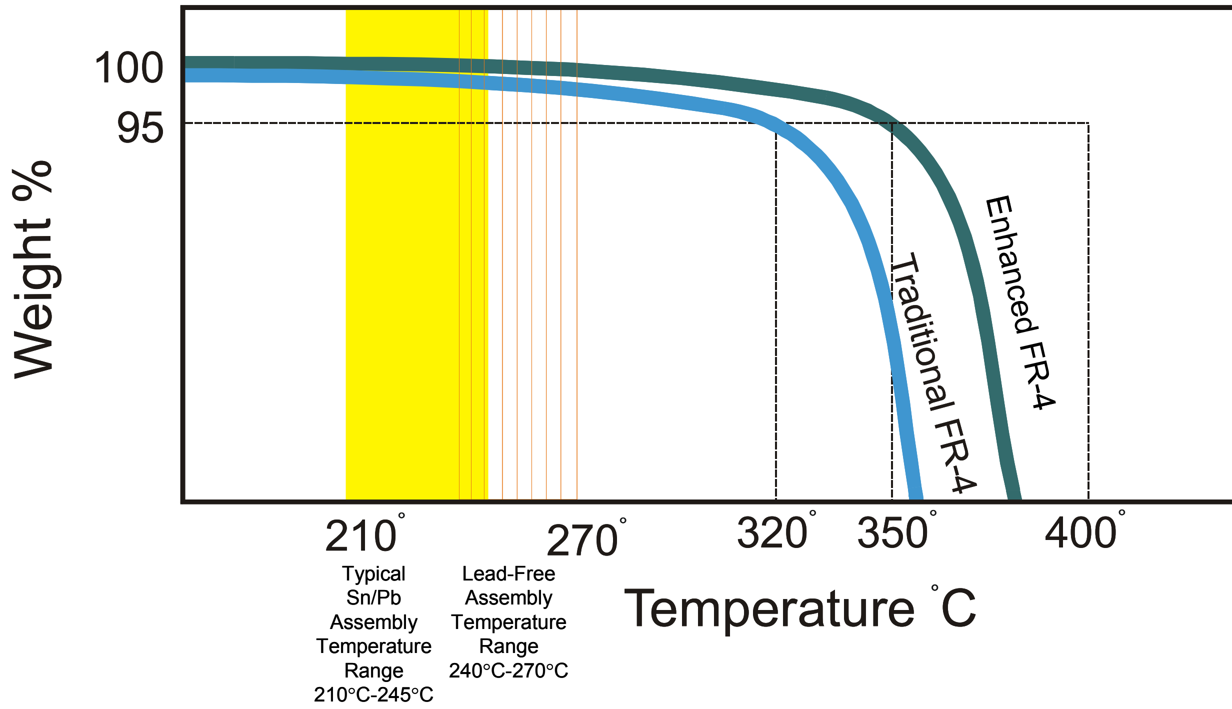

Figure 4 – Decomposition Curves for Two Different FR-4 Materials with T_g Values of 175°C

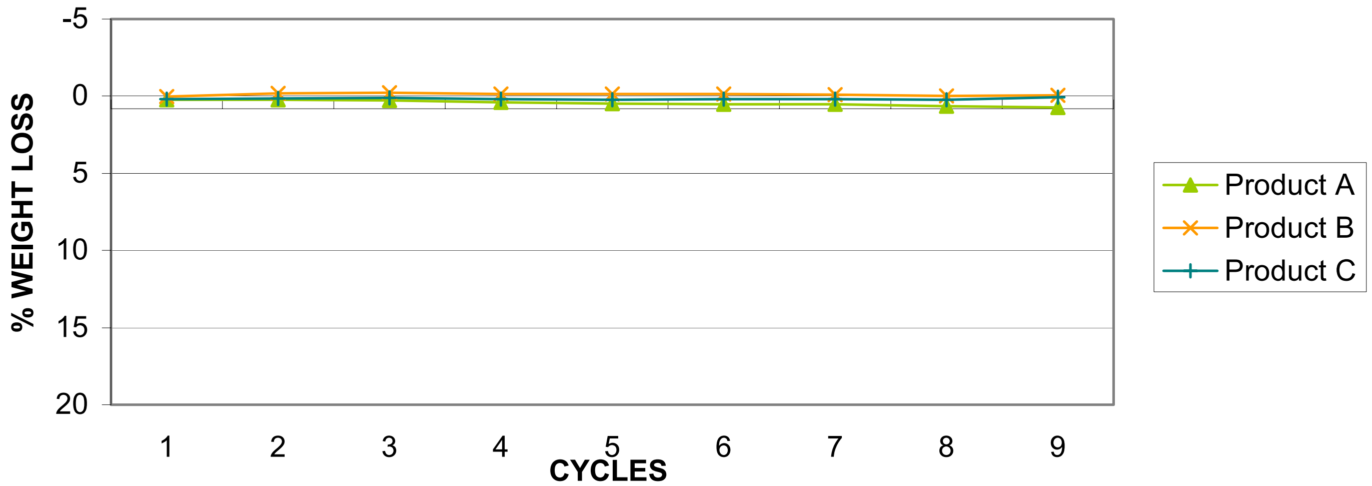

Figure 5 – Multiple TGA Cycles To A Peak Isotherm Temperature of 235°C

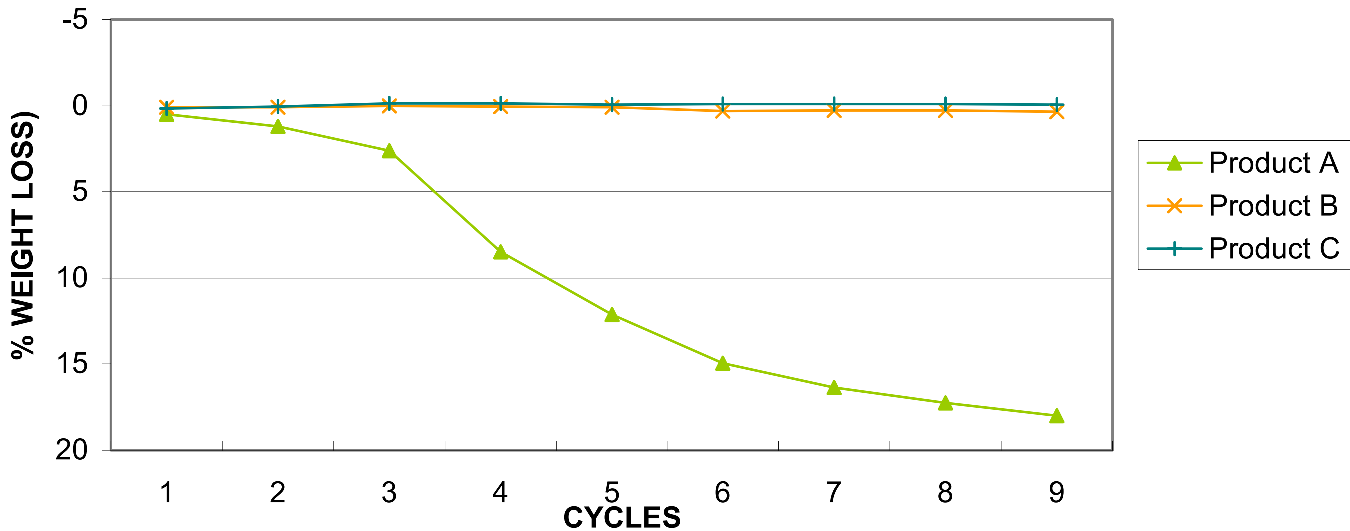

Figure 6 – Multiple TGA Cycles To A Peak Isotherm Temperature of 260°C

As evidenced in Figure 5, all four materials perform well when cycled to 235°C. Virtually no degradation is observed for any of the materials, regardless of T_g or T_d value. However, Figure 6 tells a different story. The obvious conclusion is that the materials with the higher decomposition temperatures still perform well, with little evidence of any degradation, whereas the "standard" material with the lower decomposition temperature begins to degrade significantly when exposed to multiple cycles to 260°C.

Similar thermal cycles in a dynamic mechanical analysis (DMA) unit also provides interesting results when comparing these materials. Figures 7 and 8 provide measurements of T_g by DMA through multiple cycles to 235°C and 260°C, respectively. Again, no degradation in T_g is observed when peak temperatures reach 235°C. However, with peak temperatures of 260°C, the "standard" product with a decomposition temperature of 310°C experiences a significant degradation in measured T_g upon repeated exposures. In contrast, the materials with decomposition temperatures of 350°C withstand the multiple exposures without degradation of T_g, and actually exhibit a slight increase in T_g, which based on the resin chemistry difference, is not completely surprising.

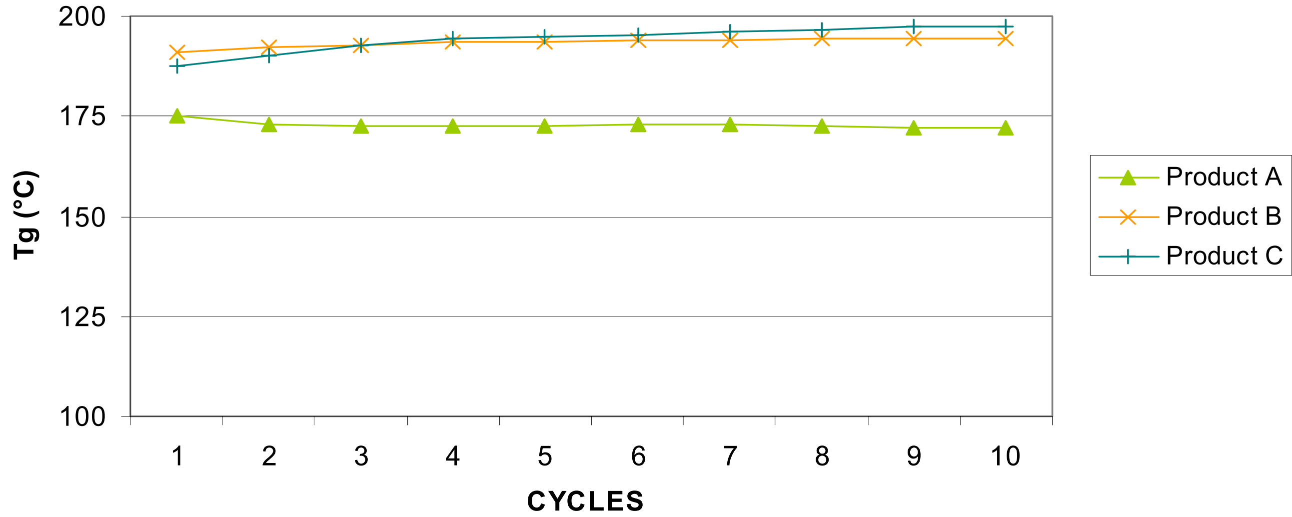

Figure 7 – Multiple DMA Cycles To A Peak Isotherm Temperature of 235°C, Measuring T_g

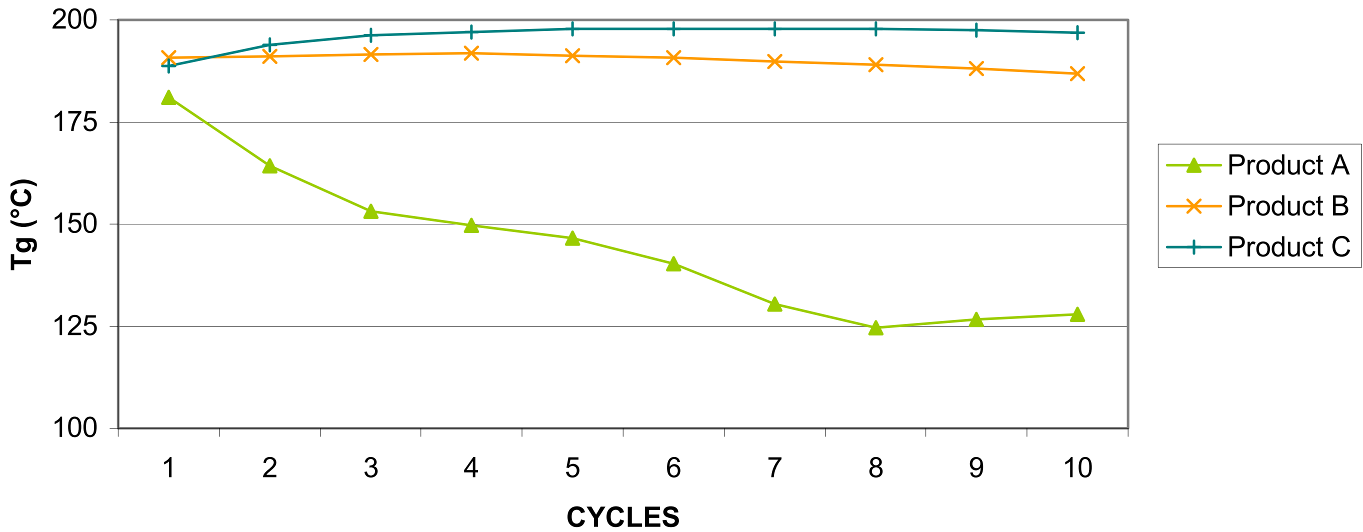

Figure 8 – Multiple DMA Cycles To A Peak Isotherm Temperature of 260°C, Measuring T_g

Another property that can be measured by DMA is storage modulus. The storage modulus is related to the flexural strength and stiffness of the material, which can be an important property to consider in assembly of PCBs. A stiffer material or one that retains its flexural strength through thermal cycles is more desirable in order to prevent sagging or warpage during assembly of components onto the multilayer board. Figures 9 and 10 present data on the storage modulus changes exhibited by products A and B when cycled to 235°C and 260°C, respectively (data on product C is being collected). In this case, the "standard" 175°C Tg material, product A, exhibits a decrease in storage modulus through multiple thermal exposures to both 235°C and 260°C, though obviously the decline is much more rapid when cycled to 260°C. Product B, the material with a 175°C T_g and a 350°C decomposition temperature retains its modulus through both 235°C and 260°C thermal cycling.

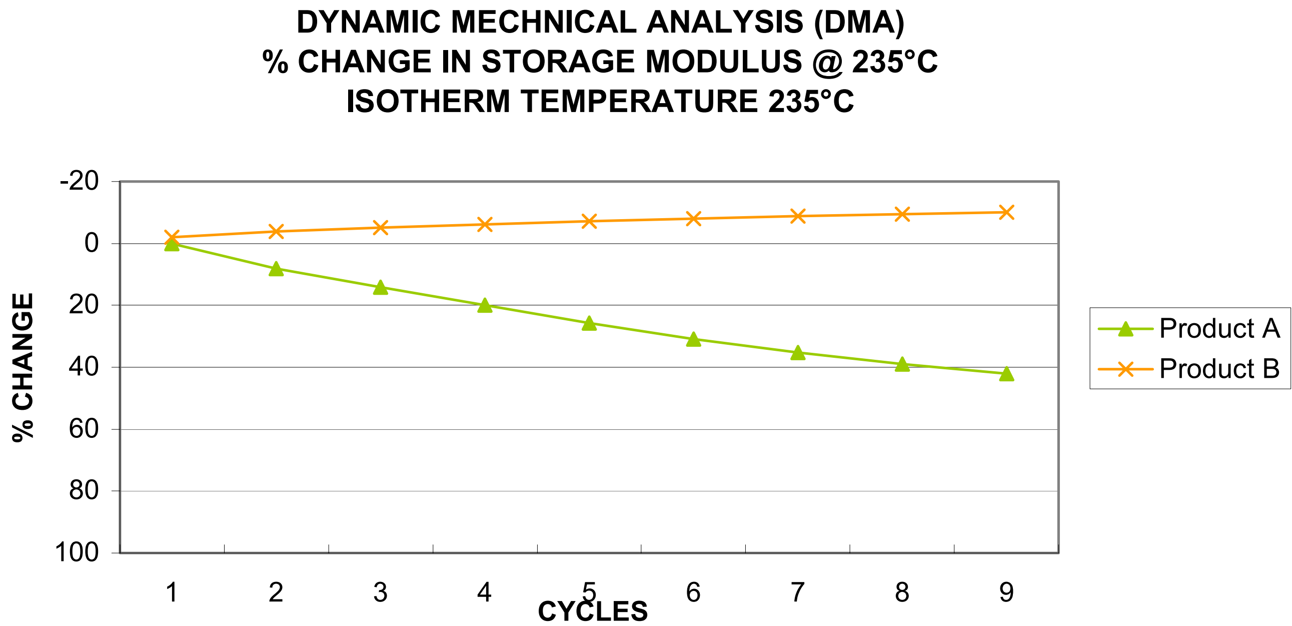

Figure 9 – Multiple DMA Cycles To A Peak Isotherm Temperature of 235°C, Measuring Storage Modulus

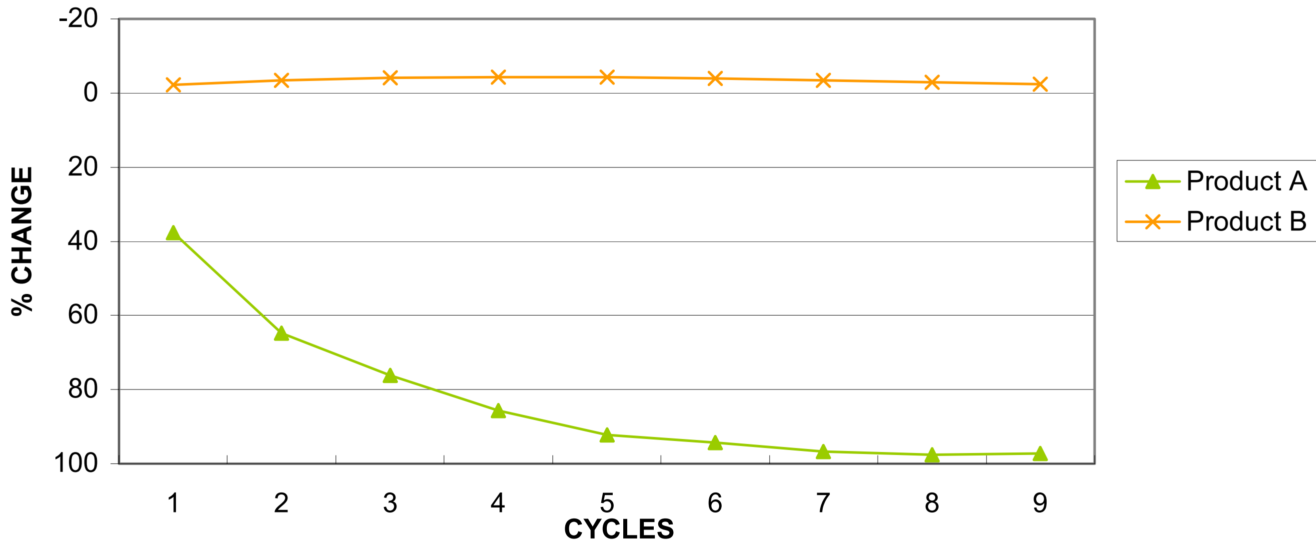

Figure 10 – Multiple DMA Cycles To A Peak Isotherm Temperature of 260°C, Measuring Storage Modulus

Assessment of Long Term Reliability

A method used to assess reliability of multilayer PCBs is the interconnect stress test (IST) procedure[vi]. The IST method uses a daisy chain plated through hole (PTH) design. The PTH's are thermally cycled to 150°C by applying an electric current to the network of plated holes and then allowing them to cool back to room temperature. Measuring how many cycles the test coupons can withstand before failure provides an indication of long-term reliability. Failure is typically defined as a 10% change in resistance of the PTH net. It is common to evaluate the performance of PCB test coupons as manufactured, or "as is" and then also evaluate performance after preconditioning of the IST test coupons performed to simulate multiple exposures though PCB assembly profiles.

In order to assess the relative effects of reduced Z-axis expansion rates and the decomposition temperature on long-term reliability, a simple comparison of four material types was performed. It should be noted however, that this was an initial screening experiment with a limited number of samples, in order to gather data to be used for larger scale tests of reliability. The four products included in this evaluation are described in Table 1.

Table 1 – Four High-Tg Materials with Different Z-Axis CTEs and Decomposition Temperatures

Material ID	Tg, °C	Pre-Tg CTE, ppm/°C	Post-Tg CTE, ppm/°C	Decomposition Temperature, °C
Product A	175	50	250	310
Modified Product A	175	45	220	310
Product B	175	50	250	350
Product C	175	45	220	350

The CTE values for these materials were measured on multiple ply 7628 laminate samples and should be used for comparative purposes. Multilayer boards built from these materials would differ based on the specific board construction chosen.

Product A is a conventional 175°C T_g FR-4 material. The "Modified Product A" has been engineered to have the same properties except for lower Z-axis CTE values. Product B exhibits comparable Z-axis CTE values to Product A, but has a decomposition temperature of 350°C, versus 310°C for Product A. Product C exhibits both reduced Z-axis CTE values and a higher decomposition temperature.

The test vehicle used for this evaluation was a 20-layer PCB with an overall thickness of 0.105" and 0.012" plated through holes. While 1.0 mil of plated copper in the holes was requested, actual measurements showed an average of only 0.7 to 0.8 mils, with isolated minimum readings down to 0.6 mils on some samples. While it is generally understood that PCB manufacturing processes can affect the results obtained on these tests, all of these samples were processed at the same time through the same processes in an effort to allow for valid base material comparisons. Figure 11 presents the average cycles to failure for these materials in the "As Is" condition as well as with preconditioning of the samples 3 times and 6 times to 230°C.

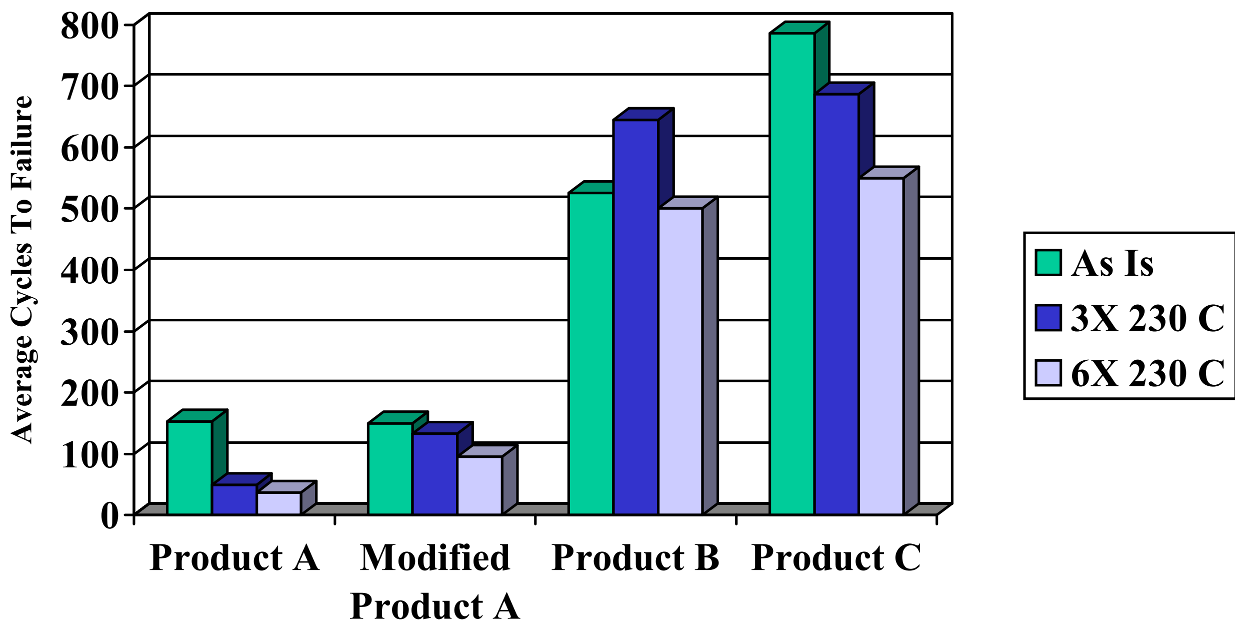

Figure 11 – IST Test Results For Four 175°C T_g FR-4 Materials

The obvious difference is between the materials with the 350°C decomposition temperatures and the materials with the 310°C decomposition temperatures. In no case did the materials with 310°C decomposition temperatures exceed an average cycles to failure value of 200. In contrast, in no case did the materials with a 350°C decomposition temperature fall below 500 average cycles to failure. While this data does suggest that the materials with reduced Z-axis expansion rates offer an improvement over the comparable materials, the improvement in this case is not nearly as large as that shown in the materials with higher decomposition temperatures. A larger scale test matrix should be designed to more fully quantify the benefits of reduced Z-axis expansion, and perhaps a larger difference in Z-axis expansion should be tested. In summary, this initial evaluation suggests that much is to be gained from a higher

decomposition temperature while a more modest improvement may be gained from reduced Z-axis expansion rates, at least in the ranges tested here.

CAF Resistance

Conductive anodic filament (CAF) formation has been the subject of much research over the years, with increased attention given to this phenomenon in recent years as circuit feature spacings are reduced[vii, viii, ix, x]. A full discussion of this phenomenon is beyond the scope of this article, however, a key finding of much of the recent work is that FR-4 materials that do not utilize dicyandiamide (dicy) as a curing agent typically exhibit improved resistance to CAF formation than comparable materials that do use dicy. Products B and C presented above do not use dicy as a curing agent and have been shown to have superior CAF resistance compared to Product A and comparable materials. Figure 12 presents data on the average insulation resistance at various hole-to-hole spacings for a 10-layer test vehicle built using Product C. Test conditions are noted in the figure.

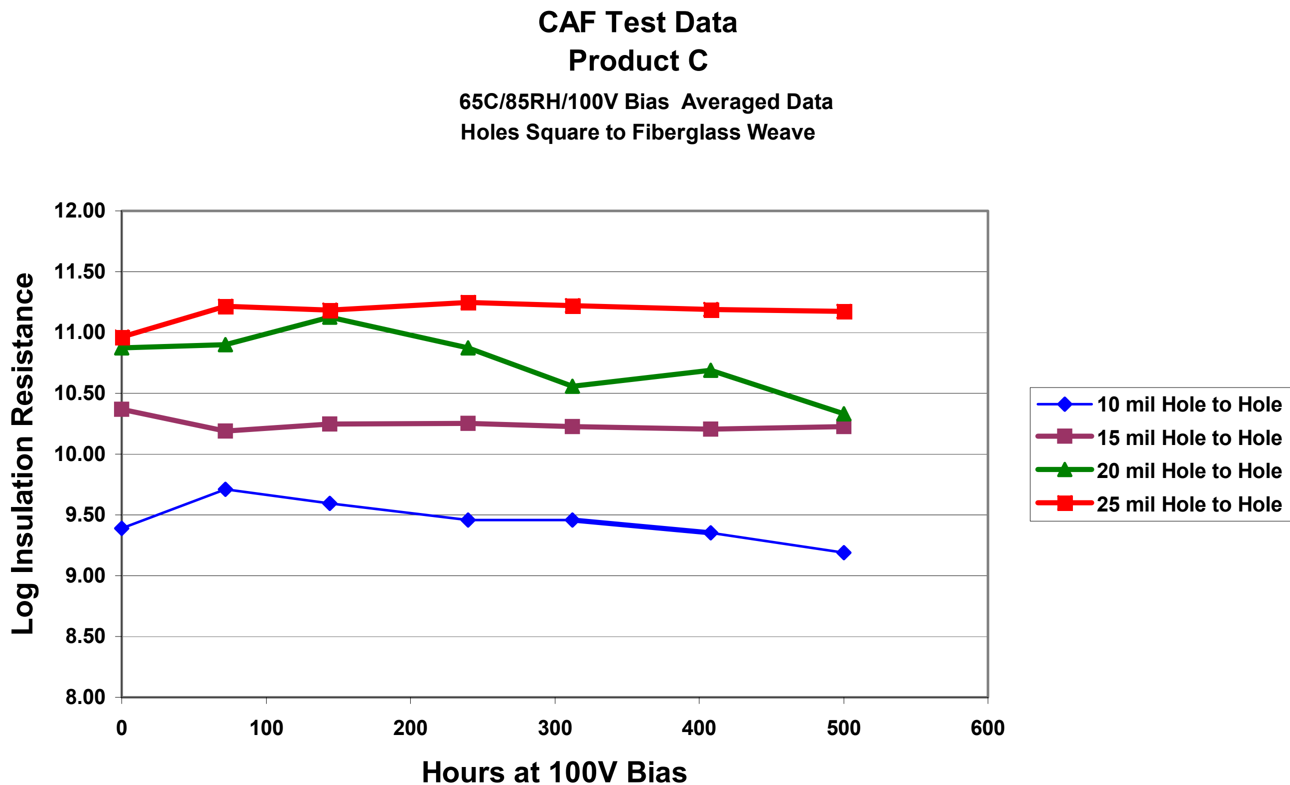

Figure 12 – CAF Test Results For Product C

Electrical Properties

One of the concerns regarding this new class of materials relates to their electrical properties, especially at higher frequencies. As noted earlier, the dielectric constant (D_k) and dissipation factor (D_f) are two critical base material properties. Re-engineering of traditional FR-4 materials should not result in significantly decreased electrical performance. A complicating factor in this discussion is the method used to measure D_k and D_f, especially at high frequencies. Table 2 presents D_k data for Products A, B, C and two competitive products using the waveguide slab method[xi, xii]. Table 3 presents D_f data for the same products. All of the samples used in these measurements were made using 2116 fiberglass cloth with 50% resin content.

Table 2 – High Frequency D_k Values For Re-engineered FR-4 Materials

Material	4.3 GHz	9.9 GHz
Product A	4.3	4.2
Product B	4.4	4.2
Product C	4.3	4.2
Competitive Product D	4.2	4.0
Competitive Product E	4.3	4.2

Table 3 – High Frequency D_f Values For Re-engineered FR-4 Materials

Material	4.3 GHz	9.9 GHz
Product A	0.015	0.017
Product B	0.020	0.022
Product C	0.017	0.018
Competitive Product D	0.024	0.024
Competitive Product E	0.017	0.018

The dielectric constant measurements show that these materials are fairly similar, with product D perhaps showing a slightly lower D_k. However, there are more significant differences in D_f. Product B and product D both show higher loss values, while products B and E exhibit more typical values in comparison to conventional FR-4 materials.

Summary & Conclusions

Trends in our industry are once again forcing us to re-engineer FR-4 materials. The primary requirements of these new materials are higher levels of thermal performance and improved CAF resistance. These improvements must be made without sacrificing performance in other areas, such as in electrical performance. While it is relatively easy to improve one or two of the required properties of a material, making simultaneous improvements in several properties while maintaining processability, all at a comparable cost, is much more difficult.

Materials such as product C described above, combine higher levels of thermal performance with improved CAF resistance without significantly altering electrical properties. Products like this will once again extend the life of FR-4.

Other conclusions that can be drawn from this work include:

- Specifying T_g may be necessary, but is insufficient for the demanding applications discussed.
- Reducing Z-axis expansion can result in improvement in measures of long term reliability, however,
- Increased decomposition temperatures appear to have an even larger impact, at least in the reliability test method described.

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