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BDX-613-2011

TESTING THE INTERLAMINATE ADHESION
OF MULTILAYER PRINTED WIRING BOARDS

By J. W. Lula

Published September 1978

Topical Report

Prepared for the United States Department of Energy
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**Kansas City
Division**

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Interlaminar adhesion of multilayer printed wiring boards can be tested effectively using thermomechanical analysis (TMA). TMA can be used to evaluate lamination cure cycles, inner layer surface preparation, prepreg conditioning techniques, and other process variables affecting the interlaminar adhesion of multilayer printed wiring boards. Because of its simplicity, TMA can also be used as a production quality control test.

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A prime contractor with the United States Department of Energy under Contract Number EY-76-C-04-0613

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SUMMARY

Thermomechanical analysis (TMA) can quantitatively measure the copper to prepreg interlaminar adhesion of an epoxy/glass multilayer printed wiring board (PWB). Adhesion is measured by noting the time a test specimen will remain at 260°C without blistering (t_{260}). The specimen is heated at 20°C/minute from room temperature up to 260°C, at which point the temperature is held constant. A sudden expansion of the specimen while at 260°C is interpreted as a blister. By recording the expansion during heat up, the glass transition temperature (T_g) also is noted. Using these TMA results, variables in the multilayer PWB manufacturing process may be evaluated as to their effect on product quality.

Tests show that TMA results are directly related to the processing and handling of the inner layers and prepreg prior to lamination. In a series of tests, a five-layer multilayer PWB made from FR-4 material with no inner layer surface treatment and no prepreg conditioning had a t_{260} of zero. When a black oxide inner layer surface treatment was used, a t_{260} of 2.5 minutes resulted. Removal of moisture from the prepreg in combination with black oxide inner layer treatment increased the t_{260} to 4.5 minutes. Moisture removal from the prepreg also increased the T_g from 124°C to 137°C. These TMA results directly correspond with the performance of production multilayer PWBs manufactured according to the respective processes.

DISCUSSION

SCOPE AND PURPOSE

A long term project has been started to develop a lamination process that will consistently produce ultrathin multilayer printed wiring boards which are free from delamination problems. As the initial phase of this project, a test was to be developed, which would quantitatively measure the interlaminar adhesion of multilayer PWBs. In addition, the test was to be such that it could be incorporated as a quality control test to monitor the interlaminar adhesion of multilayer PWBs on a regular basis.

ACTIVITY

Some multilayer PWBs manufactured at Bendix Kansas City have had a history of delamination problems. Bendix had been successfully producing other multilayer PWBs, but the multilayers with problems were of a different construction (three of the four internal conducting layers were large copper ground planes) and went through high temperature processes during manufacture (three temperature excursions $250 \pm 10^\circ\text{C}$). The delaminations were at the copper/epoxy prepreg interface and occurred following the solder dip and solder leveling processes and also during drag soldering of the next assembly. A project objective was to develop a lamination process which consistently produced quality multilayer PWBs which were not subject to delamination problems.

To measure progress in improving the interlaminar adhesion, a quantitative test was required. A literature survey and discussions with other manufacturers of multilayer PWBs failed to reveal a satisfactory test. The most common test in use is a solder dip: the multilayer is dipped in or floated on molten solder for 20 to 30 seconds. If the multilayer survives without visible delamination, it is acceptable; if not, it fails. This type of go-no go test was considered inadequate.

In the literature survey, a reference was found in which the author discussed using thermomechanical analysis (TMA) to measure the blister resistance of copper clad laminates.¹ This use of TMA suggested that TMA might be suitable for testing multilayer PWBs. Initial tests were encouraging. As the investigation proceeded, it was found that TMA will measure the degree of cure of the epoxy resin in the multilayer PWB, as indicated by the glass transition temperature (T_g). Also, TMA will give a quantitative measurement of the interlaminar adhesion, as indicated by the length of time a specimen will remain intact before blistering at 260°C (t_{260}). The test takes less than 20 minutes,

requires a small test specimen (6.3 to 7.1 mm diameter is recommended), and may be performed on an actual multilayer PWB, if desired.

Multilayer PWBs at Bendix are tested on standard TMA laboratory equipment with an expansion probe. The test specimen is prepared by punching a 7.1-mm-diameter disc from a multilayer laminate using a sheet metal hand punch. To minimize distortion of the specimen during punching, the 7.1-mm-diameter punch has been modified to smooth the face. After placing the test specimen in the fixture and lowering the expansion probe (with 10 g tray weight), the equipment is programmed for a 20°C/minute temperature rise from room temperature up to 260°C, at which point the temperature is held constant. A recording chart moving at a constant rate simultaneously records the temperature and expansion of the test specimen. Helium is flowed through the fixture during the test.

The construction of the laminate tested is shown in Figure 1. No circuitry has been etched prior to lamination, and there is full copper to prepreg adhesion at all the internal layers.

A typical TMA chart for a five-layer multilayer PWB is shown in Figure 2. Point A, the point at which the expansion curve changes slope, is the T_g of the laminate. Point B, the point at which the expansion curve levels off, corresponds to the temperature leveling off at 260°C. The sharp spike at point C is the blistering of the test specimen. The time interval between points B and C is the t_{260} of the laminate and is a measure of the adhesion between the copper and the cured prepreg.

The researcher verified TMA's sensitivity to changes in the processing of multilayer laminates. Examples of the effects of these changes are shown in Figures 3, 4, and 5. These figures are TMA curves of three different five-layer laminates similar in construction to Figure 1. To maintain full copper to prepreg interfaces, no circuitry was etched on the test panels. Only the inner layer surface treatments and prepreg conditioning were varied in the processing of the test multilayers. All were cured at 182°C and 2068 kPa for one hour with no postcure.

The multilayer described in Figure 3 had no inner layer surface treatment other than a hand scrub with cleaner followed by a thorough rinse. The prepreg was allowed to warm to room temperature, then removed from its package and used immediately. As the TMA curve on this laminate shows, the T_g is 124°C. The t_{260} is not measurable because a blister occurred at 244°C, long before the temperature reached 260°C. As an additional test, peel tests were run on 25.4-mm-wide strips, which were sheared from the laminate. When an outer copper clad layer was peeled at 90° from the rest of the laminate at the copper/prepreg interface, the result was 123 N/m width. Production multilayer PWBs, made to

Figure 1. Construction of Five-Layer Multilayer PWB

this process, will delaminate during solder dipping or drag solder. Delaminations also will occur during quality checks as the boards are cross sectioned and mounted for analysis.

Figure 4 shows the first improvement. The copper clad panels were treated with a commercial black oxide process. The prepreg was treated similar to the Figure 1 laminate. As can be seen, T_g remained unchanged. However, t_{260} is 2.5 minutes, indicating the laminate is much more resistant to delamination at 260°C . Peel strength was tested as before. A slight improvement to 158 N/m width was noted. Production multilayer PWBs made with black oxide will survive solder dipping, solder leveling, and drag soldering without delamination. Survival during the soldering processes may be related to the t_{260} as shown in Figure 4. When cross-sectioned for plated-through hole analysis though, delaminations, which may be related to the low peel strength, will occur.

Figure 5 shows the greatest improvement. In this case, in addition to the black oxide inner layer surface treatment, the prepreg was held in a vacuum for 24 hours to remove moisture. T_g has risen considerably, up to 137°C . The t_{260} has almost doubled, increasing to 4.5 minutes. The most remarkable improvement though, was in the peel strength: it increased by more than 10 times to 1751 to 4377 N/m width. Multilayers made with this process have no delamination problems, either in soldering processes or in cross-sectioning during quality analysis.

Figure 2. Typical TMA Curve for Five-Layer Multilayer PWB

TMA also was used to evaluate the effects of soldering operations on completed multilayer PWBs. A production multilayer PWB (five-layer, FR-4 material, black oxide surface treatment, no prepreg conditioning) was repeatedly subjected to our drag solder process. Following each pass across the drag solder, a test specimen was removed from the board in an area where ground planes existed. TMA results are in Table 1.

Figure 3. TMA Curve for Five-Layer Multilayer PWB: No Inner Layer Surface Treatment; No Prepreg Conditioning

The reduction in t_{260} times shows an obvious degradation of the multilayer PWB during the drag soldering passes, even though there was no visible damage to the boards.

These examples have shown that TMA is sensitive enough to quantitatively detect differences in the interlaminar adhesion of multilayer PWBs.

Figure 4. TMA Curve for Five-Layer Multilayer PWB: Black Oxide Surface Treatment; No Prepreg Conditioning

In addition, in tests currently underway, TMA is being used successfully to distinguish between different oxide coating thicknesses and also to determine the effects of long term storage of prepreg in dry nitrogen at room temperature.

Figure 5. TMA Curve for Five-Layer Multilayer PWB: Black Oxide Surface Treatment; Moisture Removed From Prepreg

ACCOMPLISHMENTS

A satisfactory test which quantitatively measures the inter-laminate adhesion of multilayer PWBs, the TMA test, has been developed. The TMA test may be used for laboratory work and for production quality control.

During the course of this work, it has been shown that multilayer PWBs which have large copper ground planes and are expected to survive several high temperature processes (237 to 260°C) must have the inner layers treated with an oxide and should have the

Table 1. Degradation of Laminate During Drag Soldering

Number of Passes	t ₂₆₀
0	2.2 minutes
1	1.4 minutes
2	0.8 minutes
3	0 minutes
4	Blistered at 236°C

moisture removed from the prepreg prior to lamination. These changes have been implemented into the Bendix manufacturing process for multilayer PWBs.

FUTURE WORK

Inner layer surface treatments are to be studied in detail. This study will involve testing various proprietary black oxide formulations as well as other means of "roughening" the inner layer surfaces.

The most effective means of moisture removal from the prepreg will be determined. A review of the incoming receiving tests and prepreg storage conditions also is planned. Changes will be made where required in the Bendix process to insure that material received and used will process acceptably.

REFERENCE

¹David Frisch and Richard Ciccarone, "Thermal Analysis for Evaluating Laminates," *Circuits Manufacturing*, Volume 17, Number 7, July, 1977, pp 54-58.

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