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**Multilayer Printed Wiring
Board Lamination**

By J. W. Lula

Published June 1980

Final Report

Prepared for the United States Department of Energy
Under Contract Number DE-AC04-76-DP00613.

**Kansas City
Division**

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BOARD LAMINATION

By J. W. Lula

Published June 1980

Final Report
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MULTILAYER PRINTED WIRING BOARD LAMINATION

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Prepared by J. W. Lula

The relationship of delamination resistance of multilayer PWBs made from GF material to manufacturing process variables was investigated. A unique quantitative test method developed during this project shows that delamination resistance is highly sensitive to material conditioning, to innerlayer surface treatment, and to post-lamination storage conditions, but is relatively insensitive to cure cycle variations.

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SUMMARY

Material conditioning, innerlayer surface treatment, and post-lamination storage conditions influence delamination resistance of multilayer printed wiring boards (PWBs) made from general purpose, fire retardant epoxy/glass material (GF). Changes in the lamination cure cycle, however, have little effect on delamination resistance.

A unique test method was used throughout this project to determine delamination resistance of multilayer PWBs. This test was developed at the start of this project and uses thermal mechanical analysis (TMA) to quantitatively measure delamination resistance at 260°C. In addition, the test measures the glass transition temperature of the resin within the PWB.

GF material used for laminating multilayer PWBs may contain as much as 0.75 percent moisture by weight. Material conditioning refers to removing this moisture and keeping the material dry before lamination. Moisture removal is required for both prepreg and innerlayers. The absence of moisture is a critical factor in manufacturing multilayer PWBs with acceptable delamination resistance.

Acceptable delamination resistance also is enhanced by innerlayer surface treatment, by which copper circuitry surfaces are converted to an oxide. This treatment passivates the copper surface and provides increased surface roughness, both of which affect the bonding to the epoxy resin in the prepreg.

Once lamination is complete, storage conditions of the multilayer PWB become important. Even when the PWB is cured, moisture rapidly absorbs into the multilayer PWB and lowers the delamination resistance. Storage conditions of 50 percent Relative Humidity (RH) at 22°C for 1 month will cause a multilayer PWB to lose 90 percent of its delamination resistance.

DISCUSSION

SCOPE AND PURPOSE

This project was initiated to solve delamination problems with ultrathin multilayer printed wiring boards (PWBs) manufactured at Bendix Kansas City. Delaminations were primarily between the copper circuitry and the prepreg, and delaminations occurred during the solder dip, solder level, and drag solder processes as well as in quality inspection during cross sectioning of plated through holes. In spite of several changes made in the manufacturing process at the time, the problem persisted. Subsequently, a long term project was planned and initiated to develop a manufacturing process that would consistently produce multilayer PWBs free from delaminations.

PRIOR WORK

Multilayer PWBs have been manufactured for several years using a process acceptable for previous multilayer boards. That process, however, does not provide the controls required for manufacturing an ultrathin multilayer PWB that has several large ground planes and is subjected to solder dip and level processes.

ACTIVITY

At the beginning of this project, there was no quantitative test for measuring delamination resistance of multilayer PWBs at soldering temperatures. (A delaminated multilayer PWB is shown in Figure 1.) The two tests commonly used by PWB manufacturers (peel test and solder float) either are not suitable for soldering temperatures or are not quantitative. Subsequently, a new test method using thermal mechanical analysis (TMA) was developed (Figure 2). After punching out a 7-mm-diameter specimen from the laminate (Figure 3), placing it into the TMA fixture (Figure 4), and programming the equipment for a 20°C/minute temperature rise to 260°C, TMA will measure the delamination resistance (t_{260}) of a multilayer PWB at 260°C. In addition, TMA will measure the resin glass transition temperature. A typical TMA curve for a multilayer PWB is shown in Figure 5. A detailed description of this test has been reported.¹

Both delamination resistance and glass transition temperature are useful in evaluating manufacturing processes for multilayer PWBs. When trying to improve these values at the start of this project, the importance of material conditioning prior to lamination was realized. Material conditioning involves both dehydration of prepreg and innerlayers as well as innerlayer surface treatment.

Figure 1. Delaminated PWB

Figure 2. TMA Equipment

Figure 3. Specimen, Punch, and Laminate

Material conditioning is essential in obtaining optimum delamination resistance in a multilayer PWB.

Prepreg Dehydration

GF prepreg for multilayer PWBs, as received from the laminate supplier, normally contains as much as 0.35 percent moisture by weight and may contain as much as 0.75 percent as allowed by MIL-P-55617. To manufacture a multilayer PWB that has acceptable delamination resistance, this moisture must be removed before lamination. Methods evaluated for moisture removal include a desiccation chamber, vacuum treatment, and dry nitrogen storage. Of these, a desiccation chamber is the slowest and least acceptable. Vacuum treatment and dry nitrogen storage remove moisture at equal rates. Dry nitrogen storage is the most convenient and cost effective for production use.

Dry nitrogen storage is simple and economical. Prepreg is hung on racks in a cabinet that is continually purged with dry nitrogen (Figure 6). This hanging separates the sheets and allows them to dry faster (Figure 7). The prepreg is removed just before lamination and after being in dry nitrogen for a minimum of 48 hours and a maximum of 1 month. The minimum time allows for removal of moisture and the maximum time prevents premature aging of the prepreg while at room temperature.²

Figure 4. Closeup of TMA Fixture

Figure 5. Typical TMA Curve for PWB

Figure 6. Dry Nitrogen Cabinet

Innerlayer Dehydration

Innerlayers contain essentially the same percentage of moisture as prepreg. And, as with the prepreg, the innerlayers must be dried. Otherwise, moisture will diffuse from the innerlayers into the prepreg during lamination, lowering the delamination resistance of the multilayer PWB. Because of the added thickness (as much as 0.35 mm, compared to 0.06 mm for prepreg), moisture removal is more difficult with innerlayers. An oven bake (6 hours at 110°C) generally is recommended.

Moisture Reabsorption

Moisture removal must be followed by appropriate precautions to prevent moisture reabsorption. Both prepreg and innerlayer, because of their large surface area/mass ratio, rapidly absorb moisture and within minutes will absorb enough to affect delami-

Figure 7. Prepreg Hanging Fixture

nation resistance. As a result, both prepreg and innerlayers must be stored in a dry environment until immediately before lamination.

Dried prepreg will absorb enough moisture from a 50 percent RH/23°C environment in 15 minutes to noticeably affect a multilayer. Figure 8 shows three multilayer PWB test panels. The top panel contains prepreg that had no moisture removal treatment. The bottom panel contains prepreg that had been vacuum treated for 24 hours. The absence of voids in the resin squeezeout corresponds to the absence of moisture in the prepreg, and shows the effectiveness of the vacuum treatment for removing moisture.

The prepreg used for laminating the center test panel had been vacuum treated for 24 hours followed by a 15-minute exposure to a 50 percent RH/23°C environment. The presence of voids in the resin squeezeout corresponds to the absorption of moisture during

Figure 8. Resin Squeezeout From Three Multilayer PWB Test Panels

this short exposure and illustrates the importance of avoiding delays after removing prepreg from the dry environment.

Innerlayers behave similarly. Their normal processing includes a 6-hour oven bake at 110°C before lamination, which drives out moisture. Unless the innerlayers are stored in a dry environment, moisture quickly will reabsorb and lower delamination resistance.

Figure 9 shows three multilayer PWB panels after lamination but before the outside circuitry has been etched. All three panels were made with prepreg that had been conditioned in dry nitrogen for 24 hours. The top panel in Figure 9 used innerlayers which had not been baked. The resin squeezeout contains a large amount of voids from moisture, and TMA shows a delamination resistance of only 1.6 minutes.

Figure 9. Resin Squeezeout After Lamination

The bottom panel in Figure 9 shows the other extreme. The innerlayers were oven baked for 6 hours at 110°C, then laminated immediately. There was no chance for moisture to reabsorb, which the absence of voids in the resin squeezeout verifies. TMA shows a delamination resistance of 6.8 minutes.

The center panel in Figure 9 used innerlayers which had been stored in a 50 percent RH/28°C environment for one week, following a 6-hour bake at 110°C. During the week, enough moisture had been absorbed to lower delamination resistance by 40 percent, to 4.1 minutes. The presence of moisture is apparent by the voids in the resin squeezeout. To maintain optimum delamination resistance, innerlayers like prepreps must be kept dry until just before lamination.

Innerlayer Surface Treatment

The initial delamination problem involved inadequate bond strength at the copper circuitry-prepreg interface. At the time, the innerlayers used had no surface treatment other than a hand scrub with pumice soap. This process adequately cleaned the copper circuitry, but epoxy does not adhere reliably even to cleaned copper. Foil manufacturers circumvent this problem by using a proprietary process to grow strong, nugget-like dendrites of copper on the foil matte surface. Then a thin film of zinc is

applied. The laminate manufacturers then bond this surface to their base material. During lamination, the heat causes the thin film of zinc to diffuse into the copper, forming brass (Figure 10). The result is a rough, passive surface to which the epoxy resin readily bonds, yielding high bond strength between the copper foil and the base laminate. This process, which promotes adhesion to the copper foil, is called TW Treatment.

Throughout this proprietary process, the opposite side of the copper foil - the drum side - has remained unchanged. The bright, smooth, pure copper presents a problem to multilayer PWB manufacturers, for this is the side they must bond. To obtain acceptable delamination resistance, the bright copper must be treated before lamination. One method of innerlayer surface treatment involves converting the copper circuitry surfaces to an oxide by immersing them in a caustic, oxidizing solution at elevated temperature. The copper oxide which is formed roughens and passivates the surface and provides the basis for acceptable bond strength.

Different thicknesses of black oxide will result in different degrees of delamination resistance. The thickness that achieves optimum delamination resistance is determined by the time of panel immersion and the solution temperature.² Optimum delamination resistance occurs after a 60 second immersion at 100°C. Longer times at lower temperatures produce equally acceptable results.

Post Lamination Storage

A multilayer PWB made from moisture free prepreg and innerlayers possesses its highest delamination resistance following the lamination cycle, because at this point moisture is at its lowest concentration within the board. Moisture absorption follows the lamination cycle beginning immediately upon exposure to the environment, and causes a decrease in delamination resistance.

This decrease has been verified by punching a quantity of TMA specimens from a multilayer test panel such as depicted in Figure 11. These specimens were then stored in various humidity environments at room temperature. Periodically, specimens were removed and tested by TMA for delamination resistance. In Figure 12, the delamination resistance (t_{260}) is plotted against time for various humidities.

Any relative humidity of 10 percent or above at room temperature causes a decrease in the original delamination resistance. Higher humidities cause a more rapid decrease. Only the samples stored in dry nitrogen showed no decrease in delamination resistance.