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EFFECTS OF THERMAL EXPANSION
ON EPOXY GLASS LAMINATES

Technological Spinoff Report

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August 1974

Prepared for the
United States Atomic Energy Commission
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**Kansas City
Division**

MASTER

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Technical Communications

**Kansas City
Division**

MASTER

EFFECTS OF THERMAL EXPANSION ON EPOXY GLASS LAMINATES

BDX-613-1171

August 1974

Prepared by E. E. Combs, D/821

A study was made of the effects of thermal expansion on epoxy glass laminates used to manufacture solder dipped and leveled printed wiring boards. Gradual or instant shock encountered during the process cycle was found to have no adverse effects on circuit board quality during normal manufacturing and rework.

THE BENDIX CORPORATION

KANSAS CITY DIVISION

KANSAS CITY, MISSOURI

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SUMMARY

During standard manufacturing or rework dipping and leveling, a printed wiring board is subjected to two thermal cycles. During testing of their resistance to such thermal shock, GH and GB laminates* endured 14 cycles before their circuitry malfunctioned--seven times more than specification limits. Electrical resistance increased almost exponentially when plotted against the thermal cycles: it did not change during the first five cycles, harmlessly increased during the second five cycles, and increased sharply to failure during the last five cycles.

All laminates darkened continuously with each successive cycle, but peel strengths either did not change or became stronger after the 15-cycle test. The Atlantic GH laminate was the only one which delaminated, while the NVF GB laminate measled slightly.

During the first heating and cooling cycle--from ambient to 500°F (260°C) and back to ambient--laminate contraction varied from 0.10 to 2.78 percent. Despite the contraction, the copper holes in some laminates were larger after this first heat-cool cycle. In that temperature range, the coefficient of linear thermal expansion of the laminates was 10 to 15 times greater than that of the circuit board's plated holes.

Decomposition temperatures for the GH laminates ranged from 554 to 656°F (290 to 345°C). This variation indicates the differences in laminates which can be caused by different combinations of resin, cure mechanism and degree of cure, and resin additives.

*GH laminates are both fire resistant (will not burn) and fire retardant (self-extinguishing). GB laminates are fire resistant, but not retardant. GE laminates are neither.

DISCUSSION

SCOPE AND PURPOSE

Before releasing a standard process on newly-designed printed wiring boards, Sandia Corporation and Bendix Kansas City agreed to a short term Bendix study to determine the expansion coefficients of existing GH, GB, and GE laminates and their attached plated-through holes. Although the study revealed that plated-through holes do not break during gradual thermal cycling, it did not examine expansion caused by sudden temperature shock--such as dipping and leveling can produce--nor did it support the multiple-temperature cycling encountered during rework and next assembly soldering.

The concern over these unanswered questions resulted in additional study under PDO 6984864, Printed Circuit Fabrication. This report answers the thermal shock and multiple cycling questions; it also provides data on nine commercially available laminates.

PRIOR WORK

BDX-613-465, Solder Leveling Applied to Manufacturing, has been concerned with some of the subjects of this report. As well, an earlier report concerning the expansion coefficient of GH laminates with plated holes, issued in 1972, has been included in this report.

ACTIVITY

Nine epoxy-glass laminate samples from seven vendors were evaluated by thermomechanical analysis (TMA) and by multiple-cycle solder dipping to determine the effect of laminate expansion on copper holes and circuit peel strength. Eight GH laminates and one GB laminate were evaluated:

- General Electric's GH FR-45 with plated hole;
- Synthane Taylor's GH Lot 1789 with plated hole, and Lot 1796;
- Atlantic Laminates' GH with plated hole;
- Mica Corporation's GH EG-823T with plated hole;
- NVF Company's GB with plated hole;

- Westinghouse's GH Lot 1019-14, and GH Lot X; and
- VOP Norplex's GH.

Laminate expansion was measured with a DuPont 941 Thermomechanical Analyzer, basically a linear variable differential transformer (LVDT). The core of the LVDT was attached to the expansion probe which contacted the sample. Changes in sample dimensions resulted in dc voltage output of the LVDT recorded on the thermomechanical analysis chart. Dimension changes as small as 0.012 mil (0.3 μ m) were detected. The samples were heated by a silver heating block which could vary the heating rate from 1 to 50°C per minute.

The laminates were cut into quarter-inch (6 mm) squares and placed under the expansion probe. Each plated hole was situated in the center of its laminate sample (Figure 1), and was placed under the expansion probe so that the 0.10-inch-diameter (2.54 mm) probe just covered the hole and the area around it. The laminates were heated at 10°C per minute. Each laminate and plated hole with laminate was heated to 260°C and then cooled to room temperature. Through this cycle, dimensional changes in the laminate were recorded.

Figure 1. Plated Hole in GB Laminate Placed Under Expansion Probe

The printed wiring boards with plated holes were repeatedly dipped in 243°C solder for 10 seconds and quenched in flux remover at ambient temperature. The same circuit pattern and hole diameters were used on each laminate sample. Peel strengths were measured before and after multi-thermal cycling (Table 1) after electrical resistances were measured on a Model 143D Micro-Ohm Meter approved for non-destructive inspection of copper thickness on production circuit boards (Table 2).

Table 1. Peel Strength (Pounds per Inch Width)

Laminate	As Made	After 15 Cycles
Synthane Taylor GH	12.17	10.79
NVF GB	*	12.30
Atlantic GH	*	10.75
GE FR-45 GH	13.23	16.40
Mica GH	11.60	12.50
Westinghouse GH	*	12.04
*Unavailable Data		

These three laminate properties were measured with the thermomechanical analyzer (Tables 3 and 4):

- Percent contraction after one cycle;
- Coefficient of linear thermal expansion (CLTE) above the glass transition temperature (T_g); and
- The T_g itself.

Table 2. Plated Hole Resistance (Microhms)

Material	Cycle Number	Hole Number				Average
		1	2	3	4	
Number 2 Synthane Taylor GH	0	048	056	316	337	189.3
	5	040	050	345	350	196.3
	15	050	076	475	504	276.3
Mica GH	0	064	063	303	316	186.3
	5	064	062	306	318	187.5
	15	147	125	534	556	340.5
Number 2 GE FR-45 GH	0	058	056	325	343	195.5
	5	055	045	312	332	186.0
	15	122	114	465	518	304.8
Atlantic GH	0	066	054	288	309	179.3
	5	044	047	300	325	179.0
	15	092	107	557	570	331.5
NVF GB	0	054	055	333	333	193.8
	5	051	053	375	338	204.3
	15	249	217	621	719	451.5

Table 2 Continued. Plated Hole Resistance (Microhms)

Material	Cycle Number	Hole Number							Average
		1	2	3	4	5	6	7	
Westinghouse GH	1	138	218	134	177	142	090	094	141.9
	5	134	212	127	169	147	080	085	136.3
	10	141	241	156	234	210	138	146	180.9
	15	170	304	183	670	792	490	566	453.6
Number 1 Synthane Taylor (White) GH	0	052	336	054	310	058	316		187.6
	4	054	361	059	317	064	316		195.1
	6	044	365	062	340	064	320		199.1
	8	062	406	062	356	081	355		220.3
	10	070	477	062	361	074	357		228.5
	12	067	503	076	421	086	385		256.3
	14	088	587	088	442	107	463		295.8
Number 1 GE FR-45 GH	0	065	386	055	292	065	327		198.3
	4	065	407	054	290	068	326		201.6
	6	073	453	055	326	076	328		218.5
	8	065	505	058	340	073	438		246.5

Table 2 Continued. Plated Hole Resistance (Microhms)

Material	Cycle Number	Hole Number						Average
		1	2	3	4	5	6	
Number 1 GE FR- 45 GH NVF GB (598921-5)	10	071	530	060	403	080	465	268.2
	12	069	580	082	457	099	467	292.3
	14	106	625	091	486	118	536	327.0
	0	066	376	058	305	057	320	197.0
	4	064	409	059	310	064	352	209.6
	6	053	410	062	332	065	378	216.6
	8	067	478	068	344	094	380	238.5
	10	081	554	077	362	076	440	265.0
	12	081	590	072	397	080	478	283.0
	14	106	697	083	410	098	543	322.8
Resistance variance between individual plated-through holes, numbered 1, 2, and so on, is related directly to their diameter. Larger resistances correspond to smaller diameter holes. More holes were available on some samples than others. The important information is how the resistance average changed rather than where the resistance started as compared to other samples.								

Table 3. Thermal Properties of Epoxy-Glass Laminates

Sample	Percent Contraction One Cycle	Coefficient of Linear Thermal Expansion Above Glass Transition Phase	Glass Transition Temperature (°C)	Thickness (μ m) (mil)
GE FR-45	0.80	170	133	62 (1.57)
NVF GB	0.41	---	122	59 (1.49)
ST Lot 1789	0.70	202	119	59 (1.49)
Atlantic Laminates	2.78	NL	116	61 (1.54)
Micaply EG 823T	0.10	220	149	61 (1.54)
ST Lot 1796	1.20	211	157	59 (1.49)
Westinghouse Lot 1019-14	0.0	233	166	60 (1.52)
Westinghouse Lot X	0.25	222	160	55 (1.39)
Norplex VOP	0.66	278	106	64 (1.62)

Table 4. Expansion Properties of Epoxy-Glass Cloth Laminates

Sample	Thickness (Inch) (mm)	Glass Transition Temperature (°C)	Coefficient of Linear Thermal Expansion From 150 to 260°C (in/in/°C) ($\mu\text{m}/\mu\text{m}/^\circ\text{C}$)	Heat-Cool Cycle	
				Shrinkage (Inch) (μm)	Percent Shrinkage
GH	0.027 (0.6)	141	0.0000146 (0.37)	0.0015 (3.8)	5.36
GH	0.043 (1.09)	130		0.00175 (44.4)	3.97
GH	0.067 (1.7)	145		0.00208 (52.8)	3.11
GH Plated Hole Only	0.091 (2.3)		0.0000146 (0.37)		
GH Hole With Lami- nate	0.091 (2.3)		0.0000177 (0.38)		
GB	0.062 (1.57)	120	0.000160 (4.0)	0.00104 (26.4)	1.70
GB	0.062 (1.57)	123	0.000148 (3.7)	0.00188 (47.7)	3.09
GB	0.062 (1.57)	115			
GB Plated Hole Only	0.067 (1.7)		0.0000147 (0.37)		
GB Hole With Laminate	0.067 (1.7)		0.0000237 (6.0)		
GE	0.061 (1.54)	110	0.000219 (5.56)	0.000905 (22.9)	1.48
GE	0.061 (1.54)	104	0.000226 (5.74)		
GE	0.061 (1.54)	110	0.000184 (4.67)		
GE Plated Hole Only	0.074 (1.87)		0.0000131 (0.33)		
GE Hole With Laminate	0.074 (1.87)		0.000203 (5.15)		

The glass transition temperature (T_g) of a polymer begins when rotations occur in the polymer chain, and the polymer changes from its rigid structure to semi-rigid or rubbery.

All of the laminates except the Westinghouse Lot 1019-14 contracted to some degree during one heat-cool cycle. The Atlantic laminates contracted 2.78 percent during one heat-cool cycle, while the other laminates contracted less than 1.20 percent.

The laminate contraction appears to be caused by the epoxy polymer undergoing a glass transition phase (T_g). During heating through the T_g , the laminates undergo several changes in physical properties. Some laminates also contract during the transition, and do not revert upon cooling. This contraction occurs only during the first heat-cool cycle.

The laminate CLTE expresses expansion in inches per inch (mm/mm) of sample thickness over a 1°C increment. The CLTE values for the laminates (Table 3) ranged from 0.00017 in./in./ $^\circ\text{C}$ ($4.3\ \mu\text{m}/\mu\text{m}/^\circ\text{C}$) for the GE FR-45 laminate to 0.000278 in./in./ $^\circ\text{C}$ ($7\ \mu\text{m}/\mu\text{m}/^\circ\text{C}$) for the Norplex VOP laminate. The CLTE of the Atlantic Laminates and NVF samples were not measured since the expansion curves were not linear above the T_g . The T_g of each laminate measured, using an expansion probe with a 50 gram load, ranged from 106°C to 166°C . This great temperature range is the result of different manufacturing processes.

A typical expansion profile of a GH laminate is shown in Figure 2. A typical TMA profile showing the T_g is shown in Figure 3.

The GE plated hole had a CLTE of 0.0000146 in./in./ $^\circ\text{C}$ ($0.3\ \mu\text{m}/\mu\text{m}/^\circ\text{C}$) when removed. The hole still in the board had a CLTE of 0.000203 in./in./ $^\circ\text{C}$ ($5.15\ \mu\text{m}/\mu\text{m}/^\circ\text{C}$) when measured above the T_g of the laminate. The expansion profile of the hole and board indicated the hole edges were being lifted or that the plated hole was being stretched because the board expanded faster than the copper hole. Such differences in expansion could cause the copper to break, leading to higher resistances and production problems.

The expansion profiles of the plated copper holes in the laminates (Figures 2 through 21) were similar to the expansion profiles of the laminates themselves: each plated hole expanded at nearly the same rate as the laminate alone. When the laminates and plated holes were cooled to ambient temperature from 260°C , the holes in the GE FR-45, Synthane Taylor Lot 1789, and Mica EC-823T had stretched 0.2, 0.14, and 0.2 percent respectively. The holes in the Atlantic and NVF laminates were not stretched because the total expansion of each was not great enough to strain the copper holes.

Figure 2. Typical Expansion Profile of a GH Laminate

Figure 3. Thermogravimetric Analysis of a Typical GH Laminate

Figure 4. Expansion Profile for GH Atlantic Laminate

Figure 5. Heating and Cooling Cycle for GH Printed Wiring Board With Sample Height of 0.027 Inch (0.6 mm)

Figure 6. Heating and Cooling Cycle for GH Printed Wiring Board With Sample Height of 0.067 Inch (1.7 mm)

Figure 7. Heating and Cooling Cycle for Printed Wiring Board With Sample Height of 0.043 Inch (1.1 mm)

Figure 8. Heating and Cooling Recycle of Printed Wiring Board With Sample Height of 0.027 Inch (0.6 mm)

Figure 9. Thermomechanical Expansion Analysis of Printed Wiring Board Recycle With Sample Height of 0.027 Inch (0.6 mm)

Figure 10. Heating and Cooling Cycle for NVF GB Laminate

Figure 11. Thermomechanical Analysis of Expansion for Heating and Cooling Cycle for GB Printed Wiring Board With Sample Height of 0.062 Inch (1.5 mm)

Figure 12. Thermomechanical Analysis of Expansion of Heating and Cooling Cycle for GB Laminate With Sample Height of 0.061 Inch (1.5 mm)

Figure 13. Thermomechanical Analysis of GE Printed Wiring Board Heating and Cooling Cycle With Sample Height of 0.061 Inch (1.5 mm)

Figure 14. Thermomechanical Analysis of Heating and Cooling Cycle for GE Printed Wiring Board With a Sample Height of 0.061 Inch (1.5 mm)

Figure 15. Thermomechanical Analysis of Expansion of Plated Holes Removed From Board

Figure 16. Thermomechanical Analysis of Expansion of Plated Hole With GB Laminate, and With Laminate Removed

Figure 17. Thermomechanical Analysis of Expansion of Plated Hole With Wiring Board GB, GE, and GH Laminate

Figure 18. Heating and Cooling Cycle of Plated Hole in Synthane Taylor Lot 1789 Laminate

Figure 19. Heating and Cooling Cycle for Plated Hole in Mica Laminate

Figure 20. Heating and Cooling Cycle for Plated Hole in General Electric Laminate

Figure 21. Thermomechanical Analysis of Expansion for Plated Hole From Synthane Taylor GH White Laminate Lot 1789

A plated hole and laminate from the Synthane Taylor Lot 1789 was heated and cooled five times. Each of the first four cycles increased the dimension of the plated hole slightly (about 0.2 percent each cycle). During the fifth cycle, the plated hole stretched about 0.62 mils or nearly one percent. Microscopic examination showed no evidence of a crack in the plated hole, but there was evidence of delamination which probably resulted in stretching of the hole or lifting of the land area around the hole. Figure 20 shows the effects of the five cycles on the plated hole in the Synthane Taylor laminate.

Thermogravimetric analysis (TGA) of the nine laminates (Table 5) found the initial decomposition temperature of the epoxy resin used to make the laminates, and calculated the amount of glass cloth each laminate contained.

Resistance was measured on the five laminates available with plated holes (Tables 6, 7, and 8). The resistance of each hole was measured at five different temperatures: lead wires were soldered to leads of the plated holes and the resistance measured at 25°C intervals. On the first attempt, resistance was measured up to 200°C; the tin-lead solder then melted. Lead wires were then silver soldered using only the GE board, which could undergo the most expansion over this temperature range. Any great increase in resistance could be attributed to a fracture of the plated hole caused by laminate expansion. The resistance from 25°C to 275°C increased only

Table 5. Thermogravimetric Analysis of Epoxy-Glass Laminates

Sample	Percent Glass Cloth	Initial Decomposition Temperature (°C)
GE FR-45	62	320
NVF GB	66	365
ST Lot 1789 White	61	300
Atlantic Laminates	62	300
Micaply EG-823T	61	300
ST Lot 1796	59	290
Westinghouse Lot 1019-14	61.5	345
Westinghouse Lot X	61	345
Norplex VOP	56	305

slightly with temperature. This increase was caused by an increase in resistance of the lead wires. No substantial increase in resistance was noted as the laminates were heated as high as 275°C.

Thermal shock's effect on plated holes (Figures 22 through 27) was calculated by comparing average plated hole resistance to dipping. Resistance was measured at 0, 5, 10, and 15 cycles with the first samples (Table 1). Raised terminals were apparent between 10 and 15 cycles. The second group of samples was measured at 0, 4, 8, 10, 12, and 14 cycles for more accuracy. The first 5 cycles did not usually increase the resistance, so they were eliminated to reduce testing time. Electrical resistance versus thermal cycles was found to be almost exponential: no change occurred at 0 to 5 cycles, but resistance gradually increased from 5 to 10 cycles and increased sharply to failure at 10 to 15 cycles.

Table 6. Resistance Measurements of Plated Holes
Versus Temperature

Sample	Resistance at Temperature (Milliohms)				
	25°C	150°C	225°C	250°C	275°C
GE FR-45	2.0	2.4	3.9	3.1	2.8
Synthane Taylor Lot 1789	1.8	2.5	3.4	3.4	3.6
Mica EG-823T	2.0	3.0	3.3	2.6	3.8
NVF GB	2.3	2.6	3.4	3.4	3.2
Atlantic Laminates	1.3	2.5	3.0	3.3	3.2

To compare these measurements, two plated holes were cut with a razor halfway across the barrel and perpendicular to the hole axis. The measurements, before and after, indicated the resistance had doubled. Infinite resistance was indicated when the holes were severed.

Failure rates varied minutely between laminate samples. Failures of the plated holes is apparently more dependent on plating thickness and plated hole geometry. Small diameter holes in relation to long barrel lengths and large terminal pads have the quickest failure rate: the large terminal pad gives more surface area for the laminate to push upward, and thus also causes a tensile break at the weakest point--in this case, the corner of the plated hole. All photographs of hole failure (Figure 28) show fatigue cracks or tensile breakage at the corners caused by repeated bending of the terminal pad at each cycle; no barrel cracking was observed on any of the samples.

All materials darkened with each dipping cycle. The Atlantic laminate was the only GH material that delaminated. The GB material measled lightly and uniformly after four cycles. Peel strength calculations for the sample circuit boards after 15 cycles indicated minimal change from initial readings taken before cycling. In some instances, the strength increased (Figures 29 through 32).

Text continued on page 39.

Table 7. Temperature Resistance Measurements
Using Tin-Lead Solder

Laminate	Resistance at Temperature (Ohms)				
	25°C	75°C	125°C	175°C	200°C
GB Number 1	0.098	0.104	0.111	0.116	380,000*
GB Number 2	0.098	0.104	0.113	0.119	0.121
GE Number 1	0.107	0.115	0.121	0.128	800,000
GE Number 2	0.107	0.117	0.123	0.130	0.134
GH Number 1 (0.027 Inch) (0.6 mm)	0.107	0.114	0.122	0.131	0.130
GH Number 2 (0.027 Inch) (0.6 mm)	0.107	0.114	0.121	0.131	249,000
GH Number 1 (0.043 Inch) (1.1 mm)	0.108	0.117	0.125	0.131	0.134
GH Number 2 (0.043 Inch) (1.1 mm)	0.108	0.118	0.125	0.131	0.135
GH Number 1 (0.067 Inch) (1.7 mm)	0.109	0.117	0.124	0.130	0.131
GH Number 2 (0.067 Inch) (1.7 mm)	0.107	0.114	0.120	0.126	192,000
*Large resistances were caused by melting.					

Table 8. Resistance at Temperature Using Silver Solder

Laminate	Resistance at Temperature (Ohms)							
	25°C	100°C	125°C	175°C	200°C	225°C	250°C	275°C
GE Number 1	0.220	0.245	0.252	0.266	0.273	0.281	0.288	0.294
GE Number 2	0.215	0.239	0.246	0.258	0.265	0.272	0.278	0.283

Figure 22. Effect of Thermal Shock on NVF GB Laminate

Figure 23. Effect of Thermal Shock on General Electric GH Laminate

Figure 24. Effect of Thermal Shock on Synthane Taylor GH Laminate

Figure 25. Effect of Thermal Shock on Mica GH Laminate

Figure 26. Effect of Thermal Shock on Atlantic GH Laminate

Figure 27. Effect of Thermal Shock on Westinghouse GH Laminate

Figure 28. Typical Corner Breaks From Plated Holes Caused by Bending Fatigue for 15 Cycles

Figure 29. Plated Hole Both In, and Removed From, GH Board

Figure 30. GH Laminate Under Expansion Probe

Figure 31. Plated Hole Removed From GH Board Placed Under Expansion Probe

ACCOMPLISHMENTS

Solder dipping and leveling can be used successfully not only in the manufacture, but in the rework of printed wiring boards. GH laminates can withstand ten thermal cycles (or five processing cycles) without altering printed wiring board quality. GB and GE laminates will tend to measle after four cycles (or two processing cycles), while the circuit quality is unaffected by up to ten cycles.

Previous findings show these advantages of solder leveling:

- Improved printed wiring board quality,
- Decreased printed wiring board fabrication flow times,
- Reduced inspection time and labor, and
- Improved next assembly production through uniform solderability and clean, close hole sizes for component insertion.

Overall cost has been reduced through simplification of the standard process used to produce printed wiring boards. Through Sandia's motivation and support, the information obtained in this study helped originate and improve the Bendix process engineering specification that governs solder dipping and leveling.

Figure 32. Temperature's Effect on Plated Hole Electrical Resistance