Analysis of Laminate Material Properties for Correlation to Pad Cratering

Carlos Morillo¹

Yan Ning¹, Michael H. Azarian¹, Julie Silk² and Michael Pecht¹ 1 University of Maryland, Center for Advanced Life Cycle Engineering (CALCE) College Park, MD 20742 USA 2 Keysight Technologies Santa Rosa, California

Abstract

Pad cratering failure has emerged due to the transition from traditional SnPb to SnAgCu alloys in soldering of printed circuit assemblies. Pb-free-compatible laminate materials in the printed circuit board tend to fracture under ball grid array pads when subjected to high strain mechanical loads. In this study, two Pb-free-compatible laminates were tested, plus one dicy-cure non-Pb-free-compatible as control. One set of these samples were as-received and another was subjected to five reflows. It is assumed that mechanical properties of different materials have an influence on the susceptibility of laminates to fracture. However, the pad cratering phenomenon occurs at the layer of resin between the exterior copper and the first glass in the weave. Bulk mechanical properties have not been a good indicator of pad crater susceptibility. In this study, mechanical characterization of hardness and Young's modulus was carried out in the critical area where pad cratering occurs using nano-indentation at the surface and in a cross-section. The measurements show higher modulus and hardness in the Pb-free-compatible laminates than in the dicy-cured laminate. Few changes are seen after reflow – which is known to have an effect -- indicating that these properties do not provide a complete prediction. Measurements of the copper pad showed significant material property changes after reflow.

Introduction

There are different tests to evaluate the susceptibility of laminates to pad crater: hot pin pull, ball pull, ball shear, drop and bending. There are standards for these tests by industrial organizations; nevertheless pad cratering and pad crater susceptibility identification is still a concern for original electronic manufacturers. None of the published studies have characterized the laminate materials in the localized area where pad cratering occurs. Meilunas et al [1] tested different methods and laminate materials with low loss to evaluate the susceptibility to cratering. They worked with low, medium and high loss, and they showed that the material with low loss had crack initiation but it did not propagate, as was the case with the other laminate materials. They stated that it is hard to tell which test is best for evaluation of pad crater susceptibility.

Wong and Silk [2] [3] used a variety of tests with different laminates, Table 1, that had different curing agents and filler under three different reflow conditions. The results of the tests showed limited agreement on rank order among the test methods, Table 2. A subset of these laminate samples is used in this study. From these earlier studies and experience, E and F samples have a higher susceptibility to pad cratering. Also, it has been consistently found that exposure to Pb-free reflows increases the susceptibility for all laminates. These expected results are compared to local material property measurements. The authors of this paper investigated whether there is a correlation between the mechanical properties of the laminate materials and the susceptibility for pad cratering.

| Laminate | Resin Type | Glass Transition Temp T _g (Centigrade) | Decomposition Temp T _d (C) | Time to delam T ₂₆₀ (Min) | Time to delam T ₂₈₈ (Min) | Z axis Expansion (50 to 260C) % |
|----------|-------------------|--|--|--|--|--|
| Α | Dicy Unfilled | 170 DSC | 300 | 10 | 2+ | 4.4 |
| В | Phenolic Unfilled | 175 DSC | 330 | >30 | >5 | 3.5 |
| С | Phenolic Filled | 180 DSC | 335 | >30 | >15 | 2.8 |
| D | Phenolic Filled | 180 DSC | 340 | 60 | 10+ | 2.8 |
| Е | Phenolic Filled | 210 DSC | 365 | 30+ | 10+ | 3.4 |
| F | Phenolic Unfilled | 190 DSC | 370 | 60+ | 10+ | 3.6 |

Table 1 - Properties of Laminates Used to Study Susceptibility to Pad Cratering

 Table 2 – Rank Order of Laminates in Various Tests

| Laminate Motorial | Hot Pin Pull | Cold Ball Pull | Ball Shear | Drop Test | 4-Point Bend |
|----------------------|--------------|----------------|------------|-----------|--------------|
| Wateria | Test | lest | lest | | WITHAE |
| Α | 1 | 1 | 1 | 1 | 1 |
| В | 2 | 3 | 2 | 1 | 1 |
| С | 3 | 4 | 2 | 2 | 1 |
| D | 4 | 2 | 2 | 3 | 1 |
| Е | 5 | 2 | 2 | 2 | 1 |
| F | 5 | 5 | 3 | 2 | 5 |

Experiment

Samples

Two of the three samples tested in this study are Pb-free compatible, cured with a phenolic curing agent, Laminates E and F. Laminate A, a non-Pb-free-compatible dicy-cured laminate that was consistently superior in the rank order for pad crater susceptibility, was also included. To evaluate the laminate materials, samples were cut from the test vehicles that were designed and tested in a previous study [2]. Two locations were selected on the test vehicle; one location is referred to as surface and the other as cross-section. The surface sample was indented on a feature with exposed epoxy, the fiducial soldermask keepout area, as shown in Figure 1. The exposed resin has similar surface topology and chemical exposures (copper etchants, etc) as near a BGA pad. Figure 2 shows the area selected for cross-section measurement. This area includes BGA pads.



Figure 1 - Area Selected for Surface Measurement



Figure 2 - Area Selected for Cross-Section Measurement



Figure 3 - Cross-Section of Printed Circuit Board

For the cross-section measurements, samples were cut, mounted in epoxy, ground and polished. Figure 3 shows the crosssection of the test vehicles, with different areas that correspond to the copper pad, epoxy and glass fiber bundle. Since it is very common to find the laminate crack initiation and propagation below the copper pad, this area was selected to take the mechanical properties measurement. In order to reduce the scattering of the mechanical properties introduced by the nanoindentation measurement, cross-section samples were polished until mirror-like. One rule of thumb in nano-indentation is the "Rule of 5%", where surface roughness should be 5% of the minimum penetration of the indent, which for this study is a minimum depth of 500 nm. The surface of cross-section samples was flat in order to avoid any variability from the sample preparation and testing. In the case of the samples that were tested at the surface of the fiducial keep out, the surfaces are rougher. This surface was not metallographically prepared, has topology from the toothy copper that was etched away and was directly exposed to the copper etchant, soldering and cleaning processes. These did not meet the Rule of 5%.

Test vehicles were separated in two groups: bare boards without reflow (as is) and after five exposures to reflow. Reflow samples follow a standard reflow production process at 260 °C \pm 5 °C peak, for 10 seconds and TAL of 62 – 69 seconds. A 5 mil stencil was used to print SAC305 solder.

Nano-indentation measurement

A nano-indenter assembled with a continuous stiffness module (CSM) and a Berkovich tip was used for the mechanical characterization. After cross-sectioning, samples were indented on the copper pad and epoxy board material near the PCB surface between the outer copper layer and the first bundle of glass. Measurements of the mechanical properties -- hardness and Young's modulus -- were acquired using the traditional Oliver and Pharr approach [4]

$$H = \frac{P}{A(h_c)} \tag{1}$$

In the case of a pyramidal indenter, such as the Berkovich tip used in this study, the indentation strain rate is the loading rate divided by the load [5]

$$\vec{\epsilon}_{i} = \frac{p}{p}$$
 (2)

Samples were indented at a constant strain rate of 0.05 s-1, at a depth of 500 nm at room temperature. At least 5 indentations were made on each sample.

Results and Discussion

In Table 3, it can be seen that moduli values for E and F samples are higher the modulus of sample A. As expected, samples with a phenolic curing agent have higher moduli – are stiffer – than the sample with dicy curing agent. It should also be noticed that in the moduli values for A and F samples there is no significant change after reflow. These samples have no fillers. Sample E contains filler and showed a decrease in the mechanical properties with reflow. It appears that fillers may result in changing modulus from the reflow process.

| Table 3 - Mechanical Properties in the Cross-Section with and without Reflow | | | | | | | | |
|--|---------------|---------------------------------|-----------------------------------|-----------------------------------|--|--|--|--|
| | Modulu | s (GPa) | Hardne | ess(GPa) | | | | |
| | As is | 5x Reflow | As is | 5x Reflow | | | | |
| E | | | | | | | | |
| Epoxy | 5.8 ± 0.3 | 5.4 ± 0.2 | $\textbf{0.44} \pm \textbf{0.02}$ | $\textbf{0.43} \pm \textbf{0.02}$ | | | | |
| Copper Pad | 126.3 ± 3.8 | 131.9 ± 6.1 | $\textbf{2.23} \pm \textbf{0.04}$ | $\boldsymbol{1.78 \pm 0.07}$ | | | | |
| F | | | | | | | | |
| Epoxy | 5.2 ± 0.2 | $\textbf{5.2} \pm \textbf{0.1}$ | $\textbf{0.38} \pm \textbf{0.1}$ | $\textbf{0.39} \pm \textbf{0.02}$ | | | | |
| Copper Pad | 137.5 ± 12 | 133.3 ± 3.8 | $\textbf{2.33} \pm \textbf{0.33}$ | $\boldsymbol{1.89 \pm 0.07}$ | | | | |
| Α | | | | | | | | |
| Epoxy | 4.8 ± 0.1 | 4.9 ± 0.1 | 0.36 ± 0.01 | 0.36 ± 0.02 | | | | |
| Copper Pad | 115.5 ± 6.2 | 130.1 ± 4.3 | $\textbf{2.19} \pm \textbf{0.29}$ | $\boldsymbol{1.97 \pm 0.08}$ | | | | |

Surface measurements, Table 4, showed lower values for these mechanical properties than in the cross-section measurements. The trends are the same for the surface and cross-section measurements: higher material properties for the phenolic-cure and more change with the filled resin. The results are more clearly illustrated in Figure 7. The relatively rough surface contributes to higher measurement variation in comparison to the cross-section measurements.

Table 4 - Mechanical Properties in the Surface of the Laminate with and without Reflow

| | Modulu | is (GPa) | Hardnes | ss (GPa) |
|---|----------------|--------------|---------------|---------------|
| | As is | 5x Reflow | As is | 5x Reflow |
| Е | 3.9 ± 0.49 | 2.9 ± 0.65 | 0.14 ± 0.01 | 0.08 ± 0.02 |
| F | 3.5 ± 1.11 | 3.7 ± 0.52 | 0.12 ± 0.04 | 0.12 ± 0.03 |
| А | 3.4 ± 0.26 | 4.2 ± 0.35 | 0.10 ± 0.03 | 0.14 ± 0.05 |

The curing process is used by manufacturers to enhance crosslinking in the epoxy; two of the most common curing agents are dicyandiamide (dicy) and phenol novolac (phenolic). It is also known that there is a weak correlation between the crosslinking of a thermosetting material and the Young's modulus [6] in the glassy state, but in this work, it is shown that an epoxy cured with a phenolic agent has a higher modulus, indicating stiffer material. This could be due to the molecular structure of the epoxy after cure.

Figure 4 shows the load and displacement curves for samples A and E, where the tip of the indenter penetrated up to 500 nm, at a specific load rate.



Figure 4 - Load and Displacement Curve for Epoxy Materials



Figure 5 - Mechanical Properties of Epoxy Material in the Cross-Section



Figure 6 - Mechanical Properties of Epoxy Material on the Surface



Figure 7 - Moduli Comparison between A and E Samples in different locations, Surface (S) and Cross-Section (CS)



Figure 8 – Mechanical Properties of Copper Pads

In Figure 8 are shown the modulus and hardness of the copper pads of samples E, F and A. The measurements on the copper pads show a surprising amount of change after reflow. The Young's modulus of copper in the as-received dicy-cured board was significantly lower than after reflow, and lower than that of the phenolic-cured samples. For all samples, the hardness decreased significantly after reflow. The authors suggest that the variation in the mechanical properties of copper pad comes from the laminate processing parameters, which are at a lower temperature for laminate A, and the effect of the reflow temperatures. These changes could affect reliability and warrant more study.



Figure 9 - Berkovich Impression on Copper Pad

Summary and Conclusions

This paper presents initial results of an effort to correlate pad cratering to the mechanical properties of laminate materials using nano-indentation. This method allows measurement in the localized resin layer where the fractures occur. Mechanical property measurements in the cross-section were higher than those made in the exposed resin on the surface, possibly due to the surface roughness. Both measurement locations showed the same trends:

It was found that phenolic-cured laminates had higher stiffness and hardness than the dicy cured laminate, as expected.

The sample that has filler in the resin showed a decrease in Young's modulus after reflow, suggesting that the filler has an effect on the hardness change.

The lack of material change for the resins without filler does not match the pad crater susceptibility test results and experience expected: there is consistently an increase in susceptibility with reflows.

The copper pad measurements showed that the hardness decreased after reflow for all samples. The as-received Young's modulus for the dicy-cured sample was significantly lower than for the Pb-free-compatible laminates and increased after reflow. This change could affect reliability of the copper.

It does not appear that Young's modulus or hardness correlate directly with pad crater susceptibility, although additional study would be helpful. The effect of lamination and reflow processes on material properties of the copper warrants additional study.

This study demonstrates that nano-indentation is a fast assessment technique to measure mechanical properties of laminate materials. This work obtains Young's modulus and hardness values in situ from a PCB as received and after reflow process, and look for a correlation between these mechanical properties and pad cratering.

References

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> ¹Center for Advanced Life Cycle Engineering (CALCE), University of Maryland, College Park, MD, 20142, USA, <u>cmorillo@calce.umd.edu</u>

> > ²Keysight Technologies Santa Rosa, CA







Introduction

 The industry is still looking for a definitive test to evaluate the susceptibility of PCB laminates to pad cratering



 This paper presents the initial results of an effort to correlate pad cratering to the mechanical properties of laminate materials using nano-indentation







Test Vehicle Description

From an earlier study:

- SAC305 solder applied with
 5 mil stencil
- Reflow 3x, 5x, 7x at 260C max
- 3 daisy-chained BGAs:
 - A-CABGA288-0.8mm-19mm-DC-LF305 (J3)
 - A-PBGA324-1.0MM-23MM-DC-LF-305 (J4)
 - SDRAM DDR2 512M-bit 667MHz
 1.8V 84-FBGA (U3)

10 layer PCB, 183mm x 127mm, 1.5 mm thick; unreflowed boards were available

Ref: Pad Cratering Susceptibility, Wong Boon San and Julie Silk, SMTAI 2012









Material Properties (of Laminates in Wong Study)

| | | | Glass | | Time to | Time to | Z axis |
|-----|-------|----------------------|--------------------------|---------------------|------------------|------------------|-------------|
| | | | Transition Decomposition | | delam | delam | Expansion |
| | | | Temp T _g | Temp T _d | T ₂₆₀ | T ₂₈₈ | (50 to 260) |
| Lar | ninat | e Resin Type | (C) | (C) | (Min) | (Min) | % |
| | А | Dicy Unfilled | 170 DSC | 300 | 10 | 2+ | 4.4 |
| | B | Phenolic Unfilled | 175 DSC | 330 | >30 | >5 | 3.5 |
| | С | Phenolic Filled | 180 DSC | 335 | >30 | >15 | 2.8 |
| | | Phenolic | | | | | |
| | D | Filled | 180 DSC | 340 | 60 | 10+ | 2.8 |
| | | Phenolic | | | | | |
| | E | Filled | 210 DSC | 365 | 30+ | 10+ | 3.4 |
| | | Phenolic | | | | | |
| | F | Unfilled | 190 DSC | 370 | 60+ | 10+ | 3.6 |

DSC: obtained using Differential Scanning Calorimetry

Ref: Pad Cratering Susceptibility, Wong Boon San and Julie Silk, SMTAI 2012







Pad Crater Susceptibility Test Results Rank Order From Previous Tests

| Laminate Material | H Pu | lot Pi ull Te | in est | Cc Pւ | old B ull Te | all st | Bal | l She Test | ear | Dro | ор Те | est | 4- Ben | Poin d w/ | t 'AE |
|----------------------|---------|------------------|-----------|----------|-----------------|-----------|-----|---------------|-----|-----|-------|-----|-----------|--------------|----------|
| А | | 1 | | | 1 | | | 1 | | | 1 | | | 1 | |
| В | | 2 | | | 3 | | | 2 | | | 2 | | | 1 | |
| С | | 3 | | | 4 | | | 2 | | | 2 | | | 1 | |
| D | | 4 | | | 2 | | | 2 | | | 3 | | | 1 | |
| E | | 5 | | | 2 | | | 2 | | | 2 | | | 1 | |
| F | | 5 | | | 5 | | | 3 | | | 2 | | | 5 | |

Ref: Pad Cratering Susceptibility, Wong Boon San and Julie Silk, SMTAI 2012; Ref: Pad Cratering Susceptibility, Testing with Acoustic Emission, Wong Boon San, Richard Nordstrom and Julie Silk IPC APEX 2014



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Effect of Multiple Reflows



Ref: Pad Cratering Susceptibility, Wong Boon San and Julie Silk SMTAI 2012







Effect of Multiple Reflows in Drop Test



This shows the negative effect of reflow on drop test results. However, relative rank order has changed and does not reflect actual experience with Laminates E and F, which show tendency to pad crater.

Ref: Pad Cratering Susceptibility, Wong Boon San and Julie Silk SMTAI 2012







Selection of Material for Nano-indentation

- Laminates A and F are the two extremes for most of the testing results.
- E is close to F: E also performs poorly in some tests; both are Pb-free compatible; E is filled, F unfilled.
- We selected laminates A, E and F for mechanical property measurement
 - these could provide information about the effect of the curing agent, fillers and their relationship to pad cratering.



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Indentation Test

"Indentation testing is a simple method that consists essentially of touching the material of interest whose mechanical properties such as elastic modulus and hardness are unknown with another material whose properties are known"



Indenter has known properties Sample has unknown properties

Indentation and Residual Impression

Ref: Fischer-Cripps, A.C. Nanoindentation Springer. 3rd Edition, 2011.







Nano-Indenter











Berkovich Indenter



Berkovich indenter. Tip made of diamond



Residual impression of a Berkovich indenter on Nickel.

Ref: A.P. Malshe, CIRP Annals - Manufacturing Technology Volume 59, Issue 2 2010 628 - 651







Samples

| Laminate | Resin Type | Glass Transition Temp T _g (C) | Decompositi on Temp T _d (C) | Time to delam T ₂₆₀ (Min) | Time to delam T ₂₈₈ (Min) | Z axis Expansion (50 to 260) % |
|----------|----------------------|--|--|---|--|--|
| E | Phenolic Filled | 210 DSC | 365 | 30+ | 10+ | 3.4 |
| F | Phenolic Unfilled | 190 DSC | 370 | 60+ | 10+ | 3.6 |
| А | Dicy Unfilled | 170 DSC | 300 | 10 | 2+ | 4.4 |

DSC: obtained using Differential Scanning Calorimetry



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Surface Location

Test Vehicle

Location of nano-indentation measurement: exposed resin, in fiducial soldermask keepout



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Cross-Section Location



Front



Glass bundles

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Berkovich Impression on Copper Pad



Zoomed image







ESEM Image of the Dicy Un-filled Sample



Berkovich impression on epoxy material



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Mechanical Properties of Epoxy Material in the Cross-section





Laminate E (filled) shows significant modulus decrease with reflow; A has slight increase.



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Mechanical Properties of Epoxy Material on the Surface



Measurement taken in **F** fiducial soldermask keepout



Larger measurement variation is due to surface roughness







Epoxy Moduli Comparison Between Different Locations





CS: Cross-section measurement between pad and glass



S: Surface measurement in fiducial keepout

- Young's moduli measured in the cross-section were higher than surface measurements.
- The surface (S) and cross-section (CS) presented the same behavior: Laminate E decreased with reflow, Laminate A increased with reflow.



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Mechanical Properties of the Copper Pad





- Copper on the dicy-cured laminate A had a significantly lower Young's modulus as-received.
- For all samples, the copper hardness decreased after reflow.







Summary

- Mechanical properties in the cross section of Pb-freecompatible samples have higher values than for the dicy cured non-compatible sample.
- Young's moduli of unfilled samples did not change after reflow; modulus of the filled sample decreased after reflow.
- The mechanical property values measured in the cross-section were consistently higher than those made on the surface in the same sample, however the trend with reflows was consistent.
- Surface measurements have greater measurement variation, likely due to surface roughness or chemical exposure.
- Hardness of the copper pad decreased after reflow for all samples. Copper modulus on unreflowed dicy-cured laminate was lower than with all other samples.







Conclusions

- Young's Modulus and hardness measurements show the higher values expected for the Pb-free compatible materials.
- These material properties do not appear to correlate strongly with pad crater susceptibility; the degradation expected with reflows is not evident in all samples.
- The difference between cross-section and surface results is likely due to roughness in the unmodified surface sample, and needs further investigation.
- The change in mechanical properties of the copper is interesting and warrants more study.



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Questions?