

Analysis of Laminate Material Properties for Correlation to Pad Cratering

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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.

Table 1 - Properties of Laminates Used to Study Susceptibility to 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) %
A	Dicy Unfilled	170 DSC	300	10	2+	4.4
B	Phenolic Unfilled	175 DSC	330	>30	>5	3.5
C	Phenolic Filled	180 DSC	335	>30	>15	2.8
D	Phenolic Filled	180 DSC	340	60	10+	2.8
E	Phenolic Filled	210 DSC	365	30+	10+	3.4
F	Phenolic Unfilled	190 DSC	370	60+	10+	3.6

Table 2 – Rank Order of Laminates in Various Tests

Laminate Material	Hot Pin Pull Test	Cold Ball Pull Test	Ball Shear Test	Drop Test	4-Point Bend with AE
A	1	1	1	1	1
B	2	3	2	1	1
C	3	4	2	2	1
D	4	2	2	3	1
E	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.

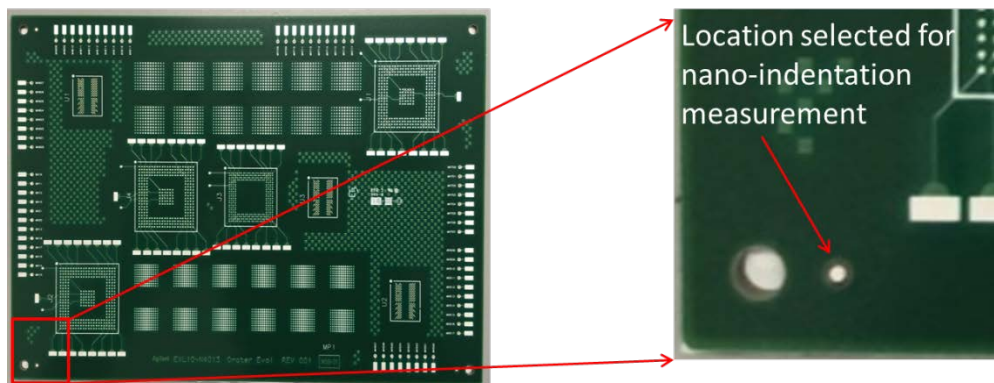


Figure 1 - Area Selected for Surface Measurement

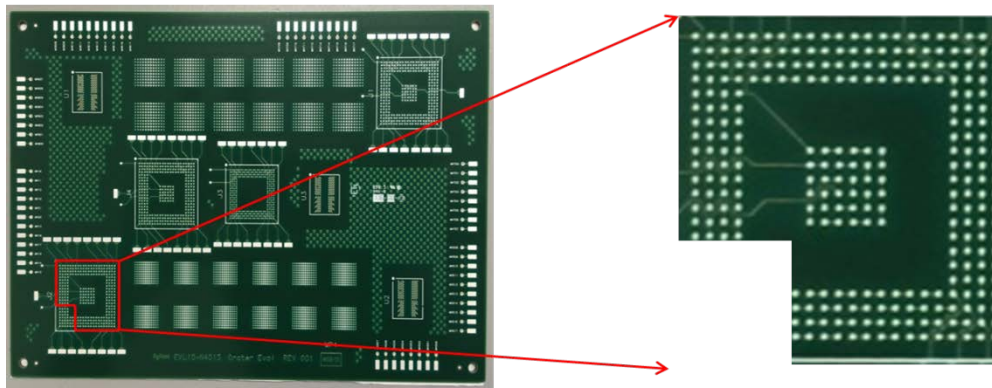


Figure 2 - Area Selected for Cross-Section Measurement

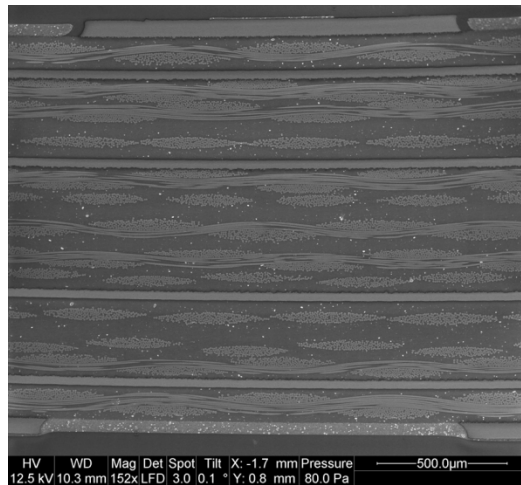


Figure 3 - Cross-Section of Printed Circuit Board

For the cross-section measurements, samples were cut, mounted in epoxy, ground and polished. Figure 3 shows the cross-section 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 nano-indentation 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 $260\text{ °C} \pm 5\text{ °C}$ 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]

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

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]

$$\epsilon_i = \frac{P}{P} \quad (2)$$

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

	Modulus (GPa)		Hardness(GPa)	
	As is	5x Reflow	As is	5x Reflow
E				
Epoxy	5.8 ± 0.3	5.4 ± 0.2	0.44 ± 0.02	0.43 ± 0.02
Copper Pad	126.3 ± 3.8	131.9 ± 6.1	2.23 ± 0.04	1.78 ± 0.07
F				
Epoxy	5.2 ± 0.2	5.2 ± 0.1	0.38 ± 0.1	0.39 ± 0.02
Copper Pad	137.5 ± 12	133.3 ± 3.8	2.33 ± 0.33	1.89 ± 0.07
A				
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	2.19 ± 0.29	1.97 ± 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

	Modulus (GPa)		Hardness (GPa)	
	As is	5x Reflow	As is	5x Reflow
E	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
A	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.

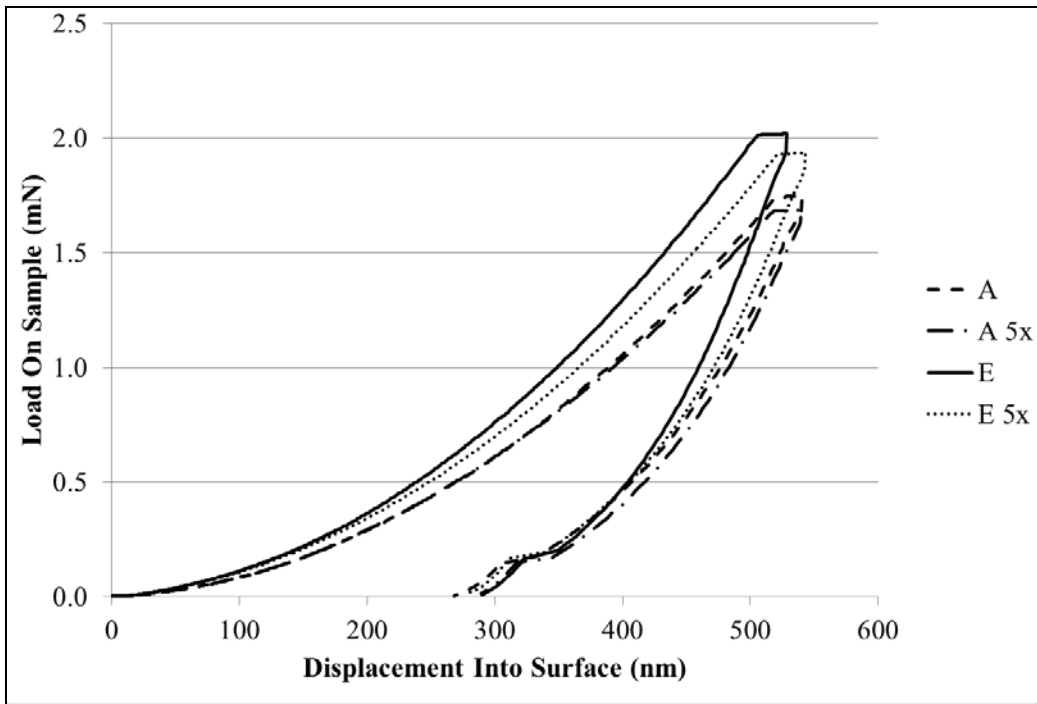


Figure 4 - Load and Displacement Curve for Epoxy Materials

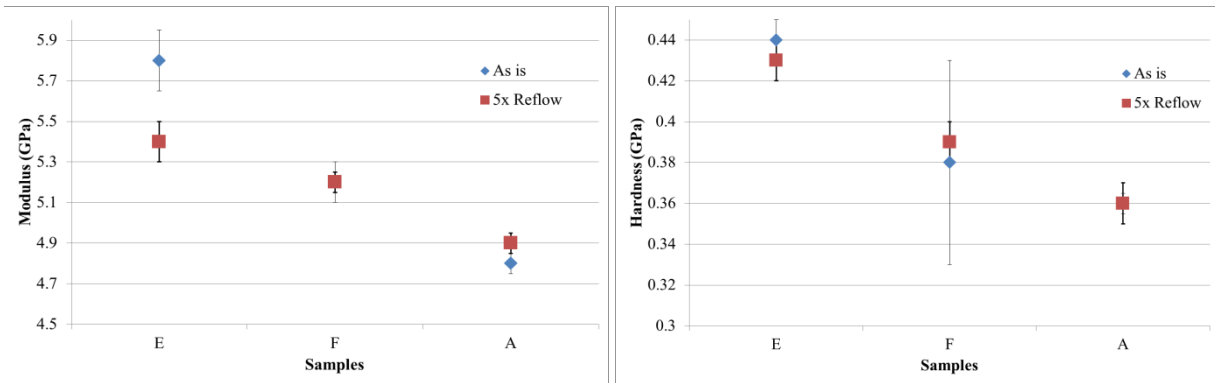


Figure 5 - Mechanical Properties of Epoxy Material in the Cross-Section

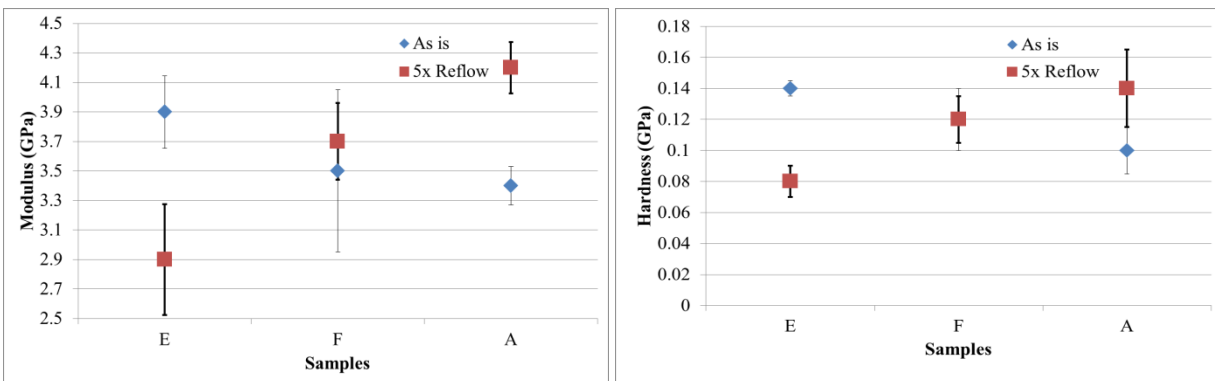


Figure 6 - Mechanical Properties of Epoxy Material on the Surface

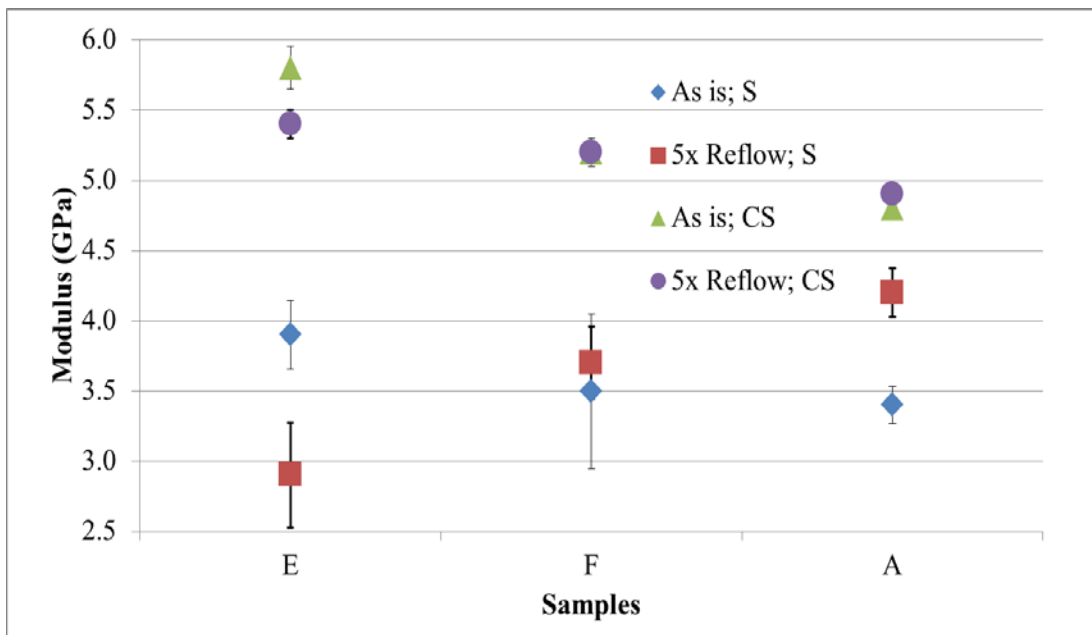


Figure 7 - Moduli Comparison between A and E Samples in different locations, Surface (S) and Cross-Section (CS)

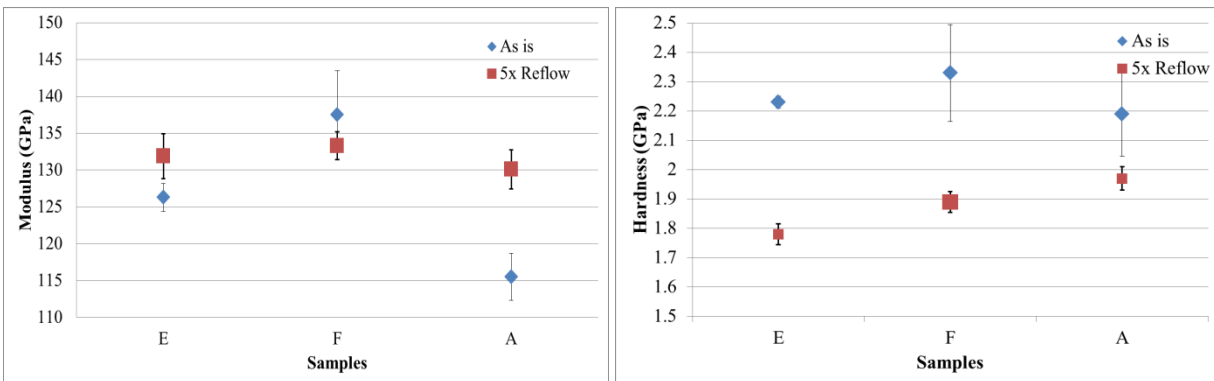


Figure 8 – Mechanical Properties of Copper Pads

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.

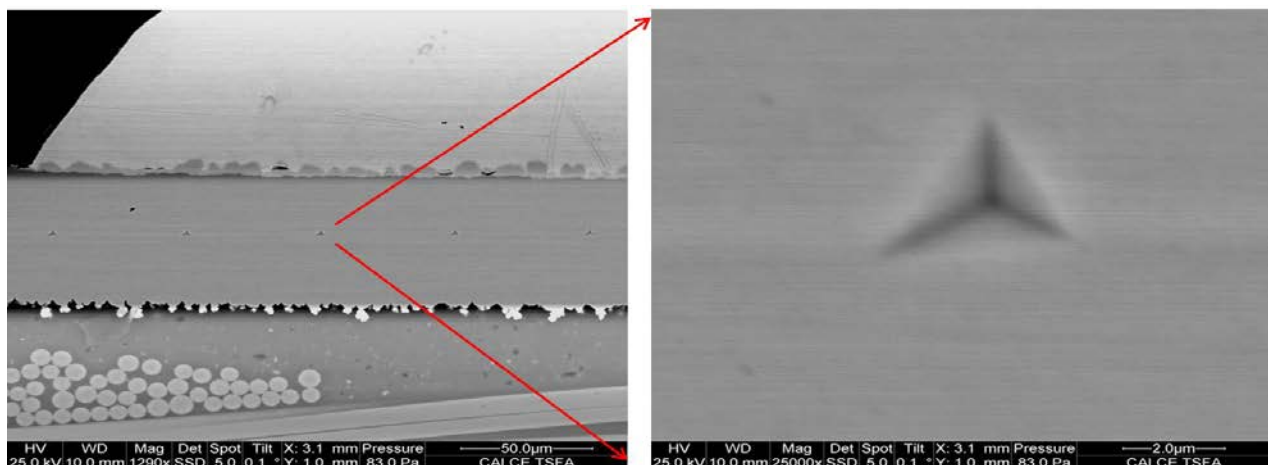


Figure 9 - Berkovich Impression on Copper Pad

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

- [1] M. Meilunas and M. Anselm, "A.R.E.A. - Low Loss Laminate Material Pad Cratering Resistance," in SMTA International, 2014, pp. 401–405.
- [2] Wong, Boon-San and Julie Silk, "Pad Cratering Susceptibility," in SMTA International 2012 Proceedings, 2012.
- [3] Wong, Boon-San, Richard Nordstrom and Julie Silk, "Pad Cratering Susceptibility With Acoustic Emission", in IPC/APEX 2014 Proceedings, 2014
- [4] W. Oliver and G. Pharr, "An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments," J. Mater. Res., vol. 7, no. 6, 1992.
- [5] B. Lucas and W. Oliver, "Indentation power-law creep of high-purity indium," Metall. Mater. Trans. A, vol. 30, no. March, 1999.
- [6] T. Kamon and H. Furukawa, Epoxy Resins and Composites IV, vol. 80. Berlin, Heidelberg: Springer Berlin Heidelberg, 1986.



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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

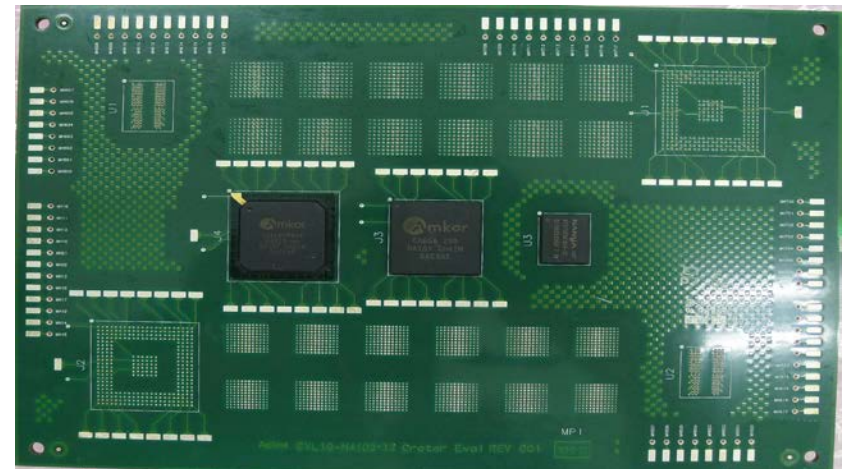




Test Vehicle Description

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



Material Properties (of Laminates in Wong Study)

Laminate	Resin Type	Glass Transition Temp T_g (C)	Decomposition Temp T_d (C)	Time to delam T_{260} (Min)	Time to delam T_{288} (Min)	Z axis Expansion (50 to 260) %
A	Dicy Unfilled	170 DSC	300	10	2+	4.4
B	Phenolic Unfilled	175 DSC	330	>30	>5	3.5
C	Phenolic Filled	180 DSC	335	>30	>15	2.8
D	Phenolic Filled	180 DSC	340	60	10+	2.8
E	Phenolic Filled	210 DSC	365	30+	10+	3.4
F	Phenolic 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

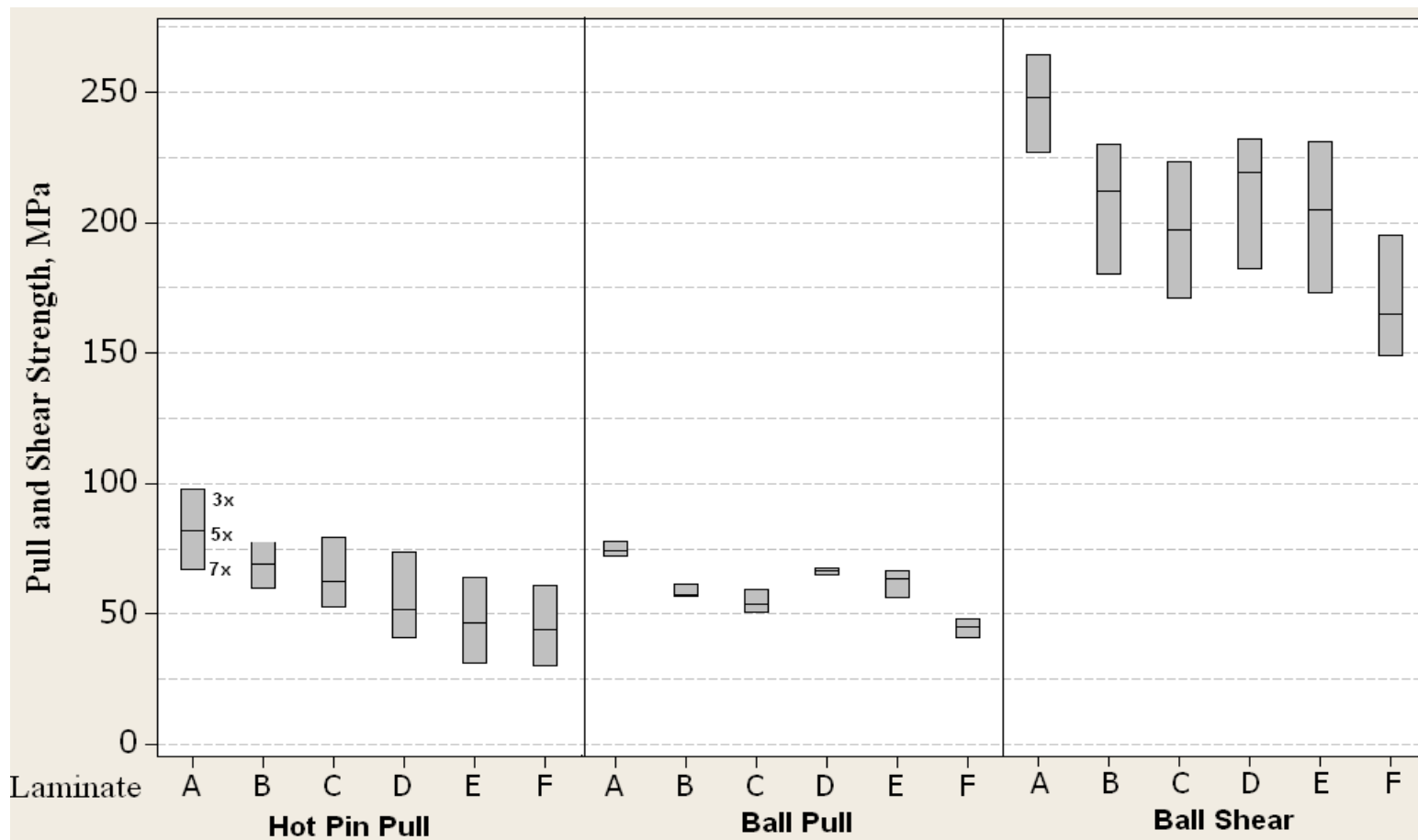
Rank Order From Previous Tests

Laminate Material	Hot Pin Pull Test	Cold Ball Pull Test	Ball Shear Test	Drop Test	4-Point Bend w/AE
A	1	1	1	1	1
B	2	3	2	2	1
C	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



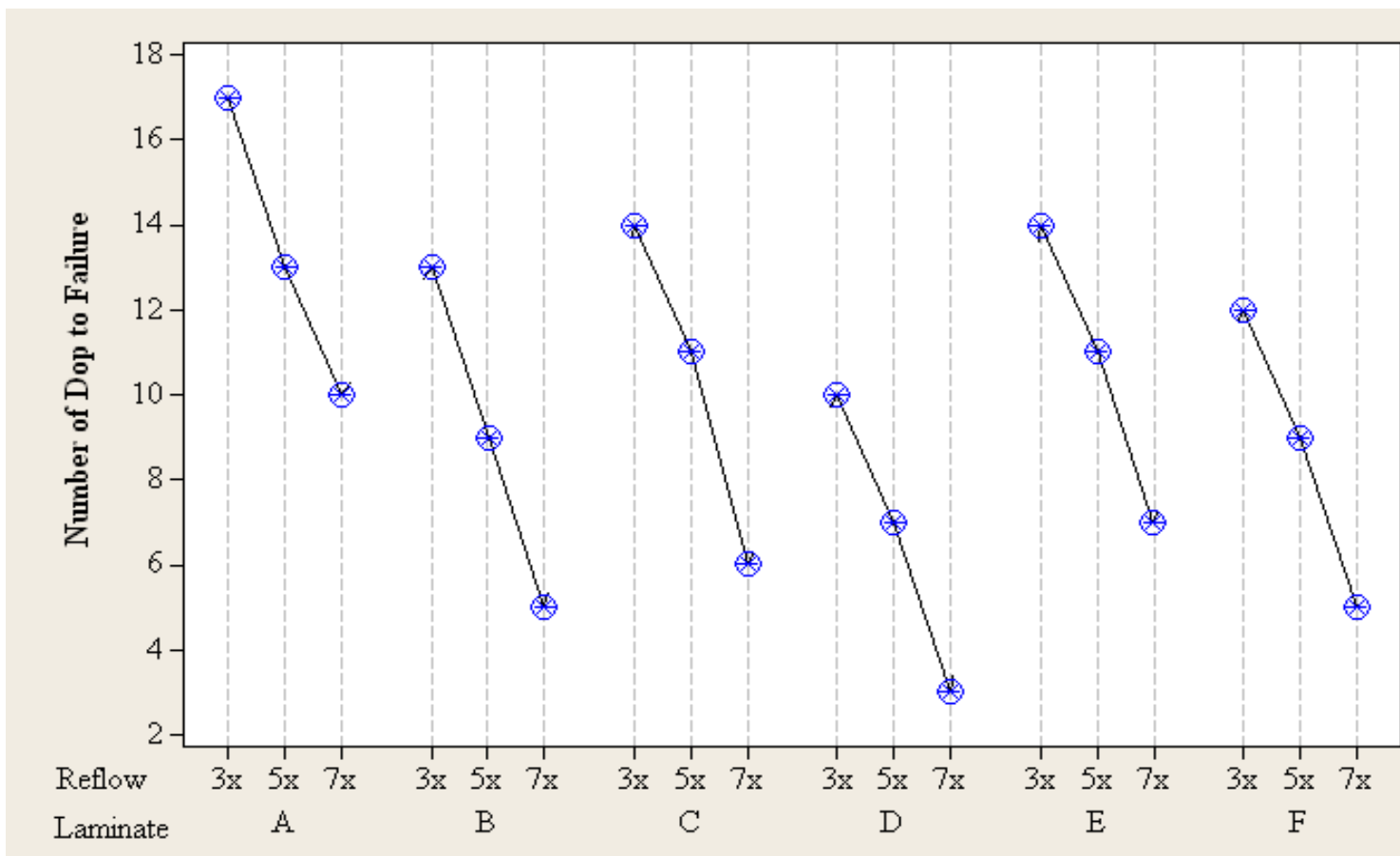
Effect of Multiple Reflows



Poorer performance after multiple reflows is expected. Ball Pull test does not reflect this.



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.



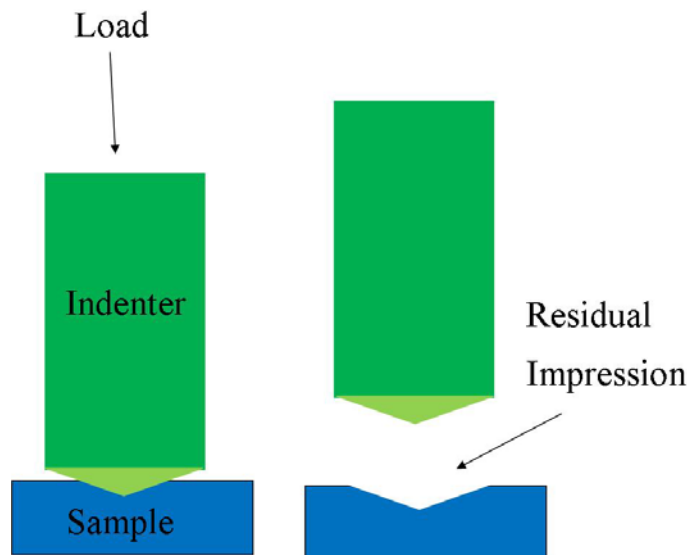
Selection of Material for Nano-indentation

- 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.



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”

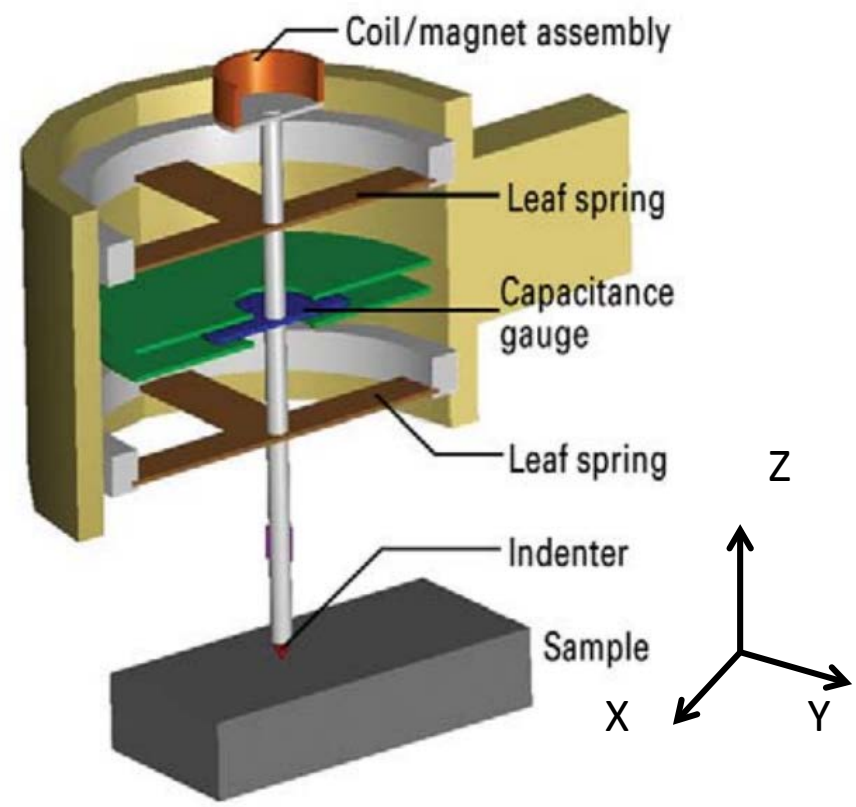


Indenter has known properties
Sample has unknown properties

Indentation and Residual Impression

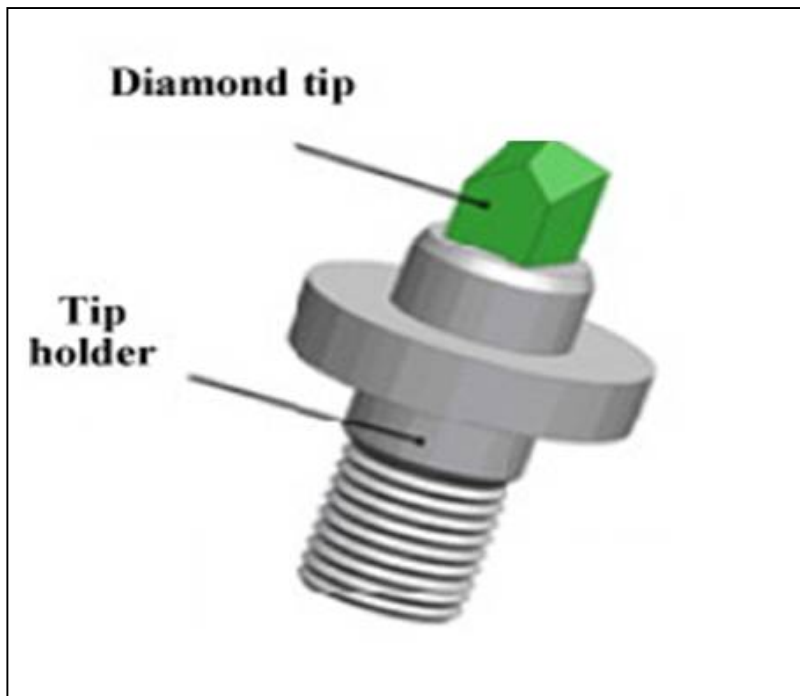


Nano-Indenter

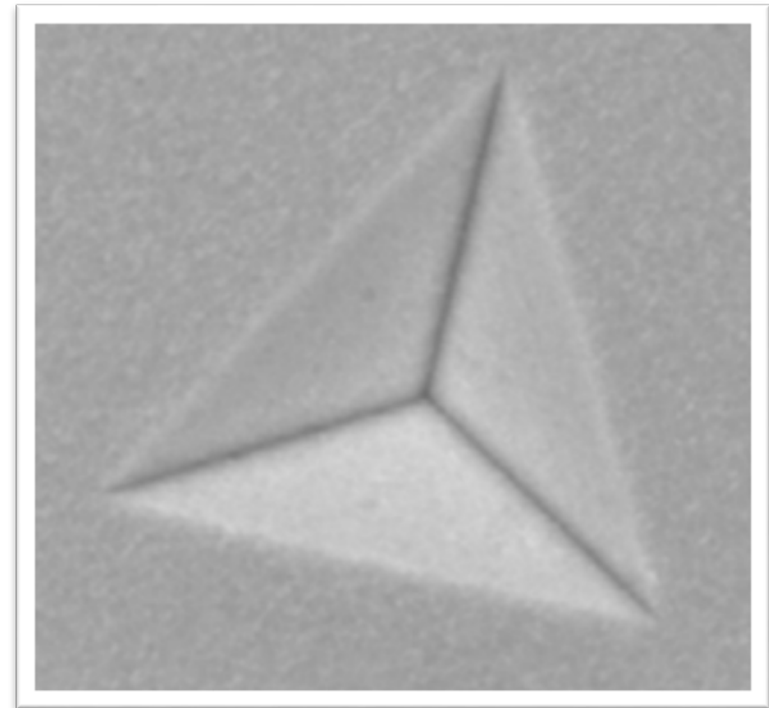




Berkovich Indenter



Berkovich indenter. Tip made of diamond



Residual impression of a Berkovich indenter on Nickel.



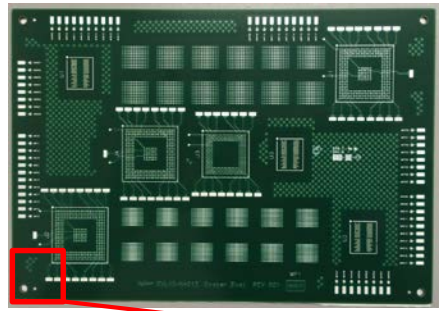
Samples

Laminate	Resin Type	Glass Transition Temp T_g (C)	Decomposition 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
A	Dicy Unfilled	170 DSC	300	10	2+	4.4

DSC: obtained using Differential Scanning Calorimetry

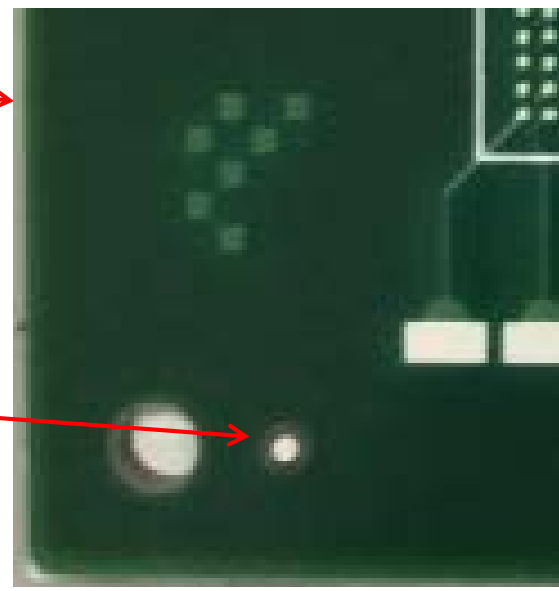


Surface Location



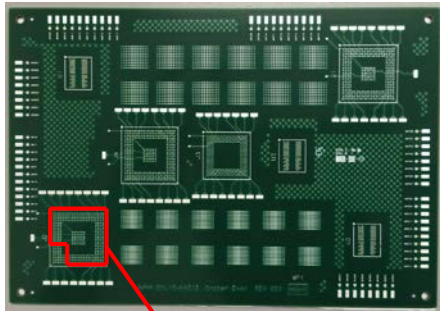
Test Vehicle

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





Cross-Section Location



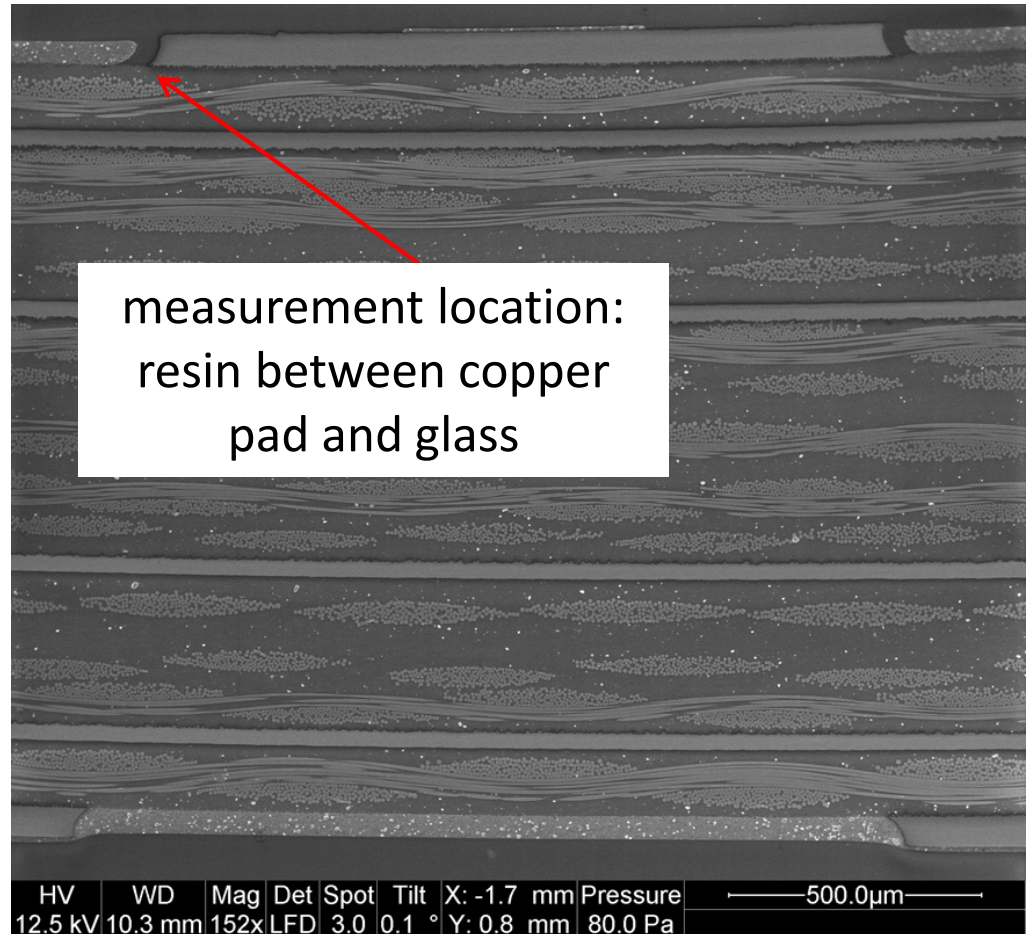
Test Vehicle



Front



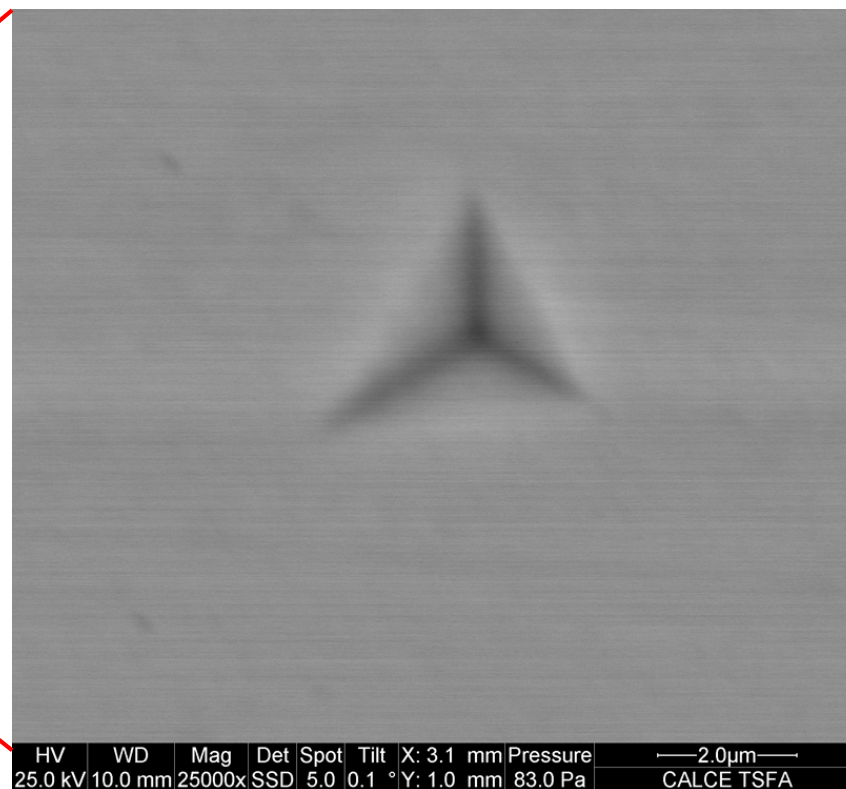
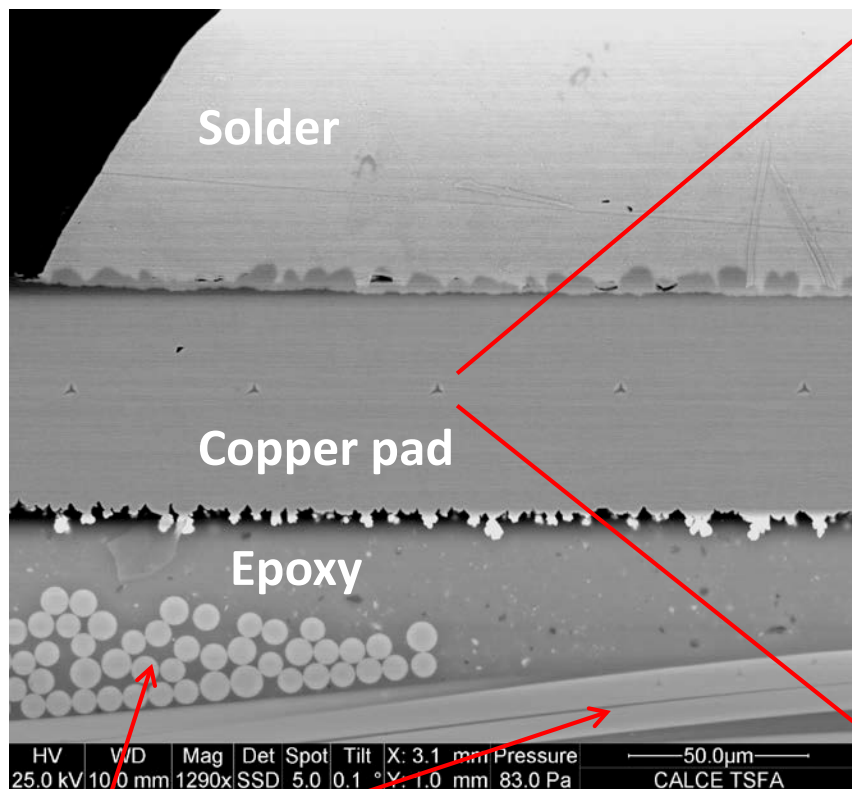
the
cross-
sectional
view





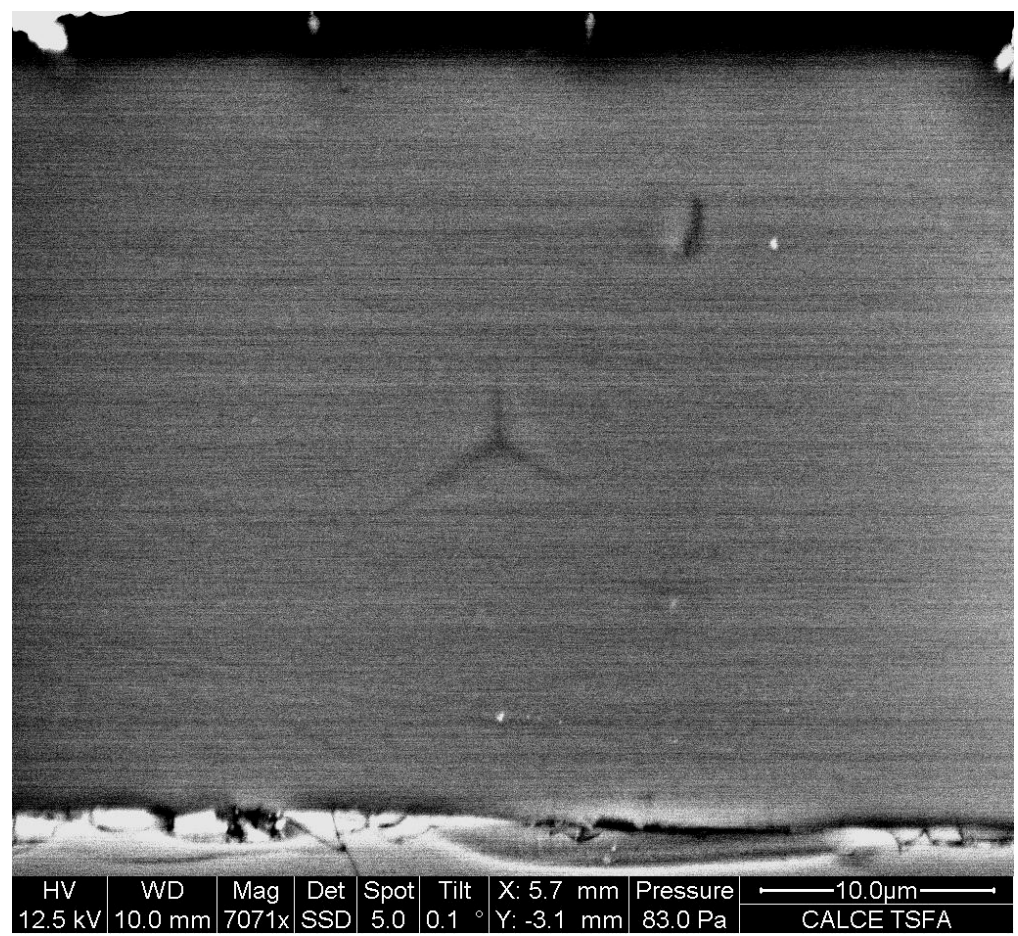
Berkovich Impression on Copper Pad

Zoomed image





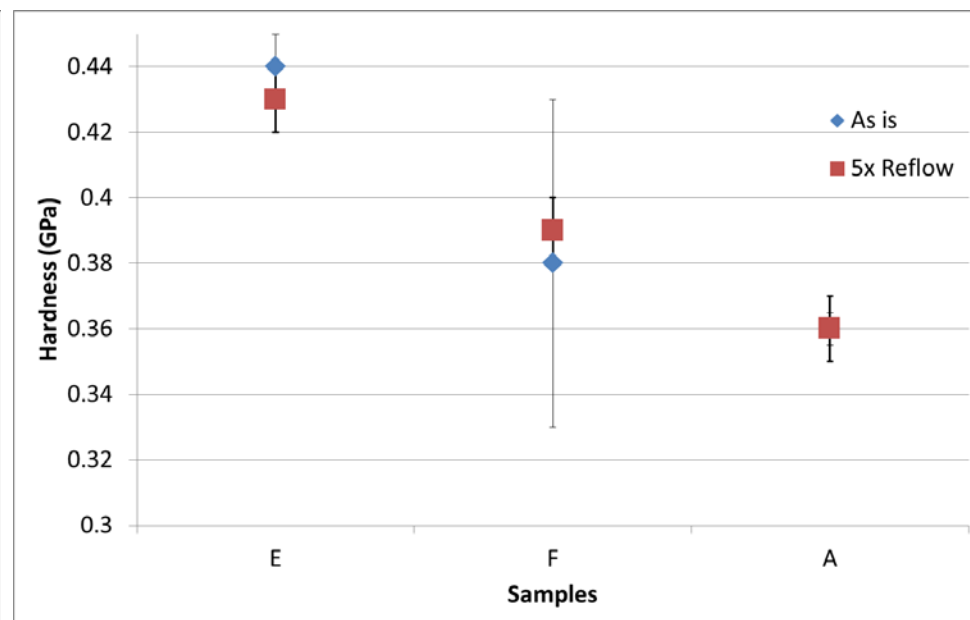
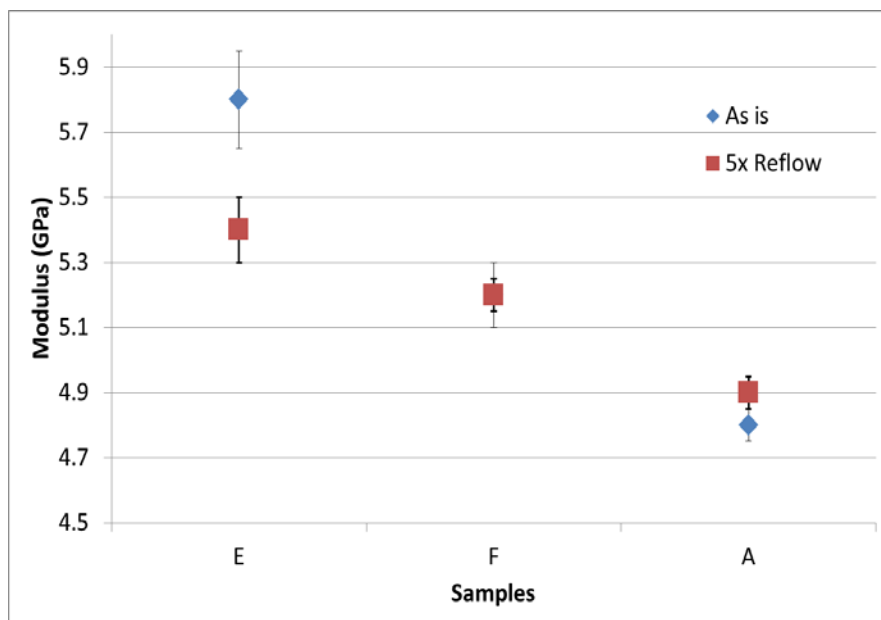
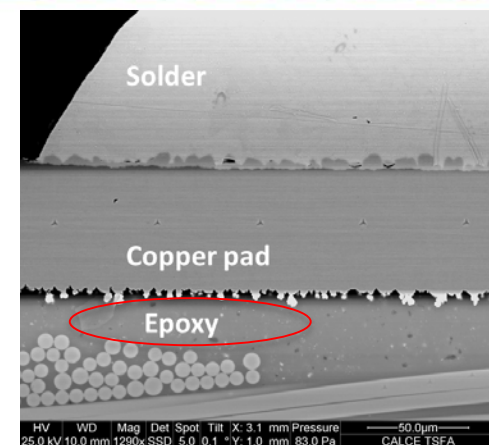
ESEM Image of the Dicy Un-filled Sample



Berkovich impression on epoxy material



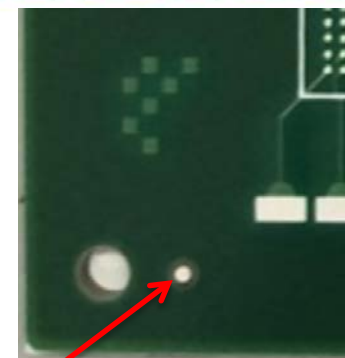
Mechanical Properties of Epoxy Material in the Cross-section



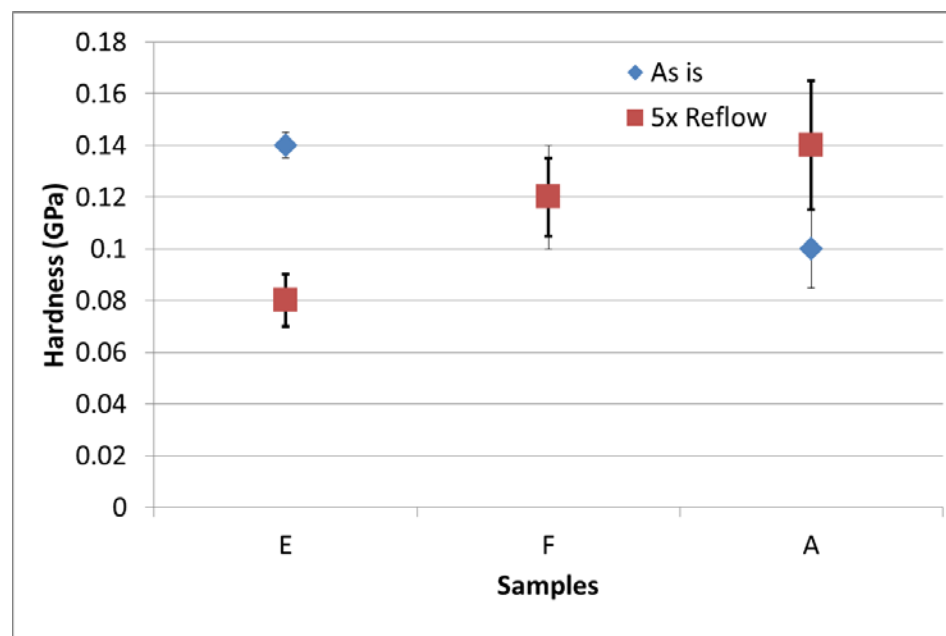
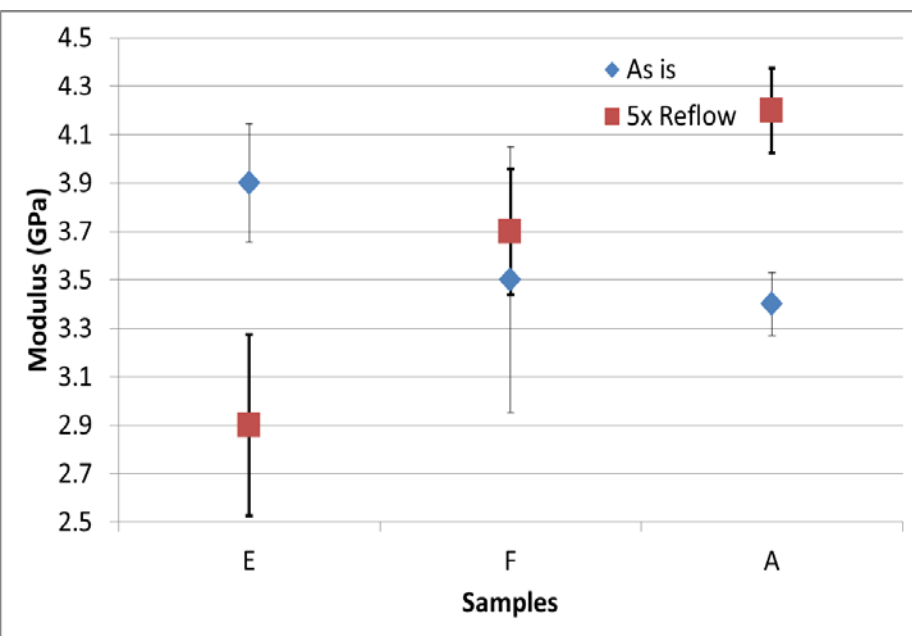
Laminate E (filled) shows significant modulus decrease with reflow; A has slight increase.



Mechanical Properties of Epoxy Material on the Surface



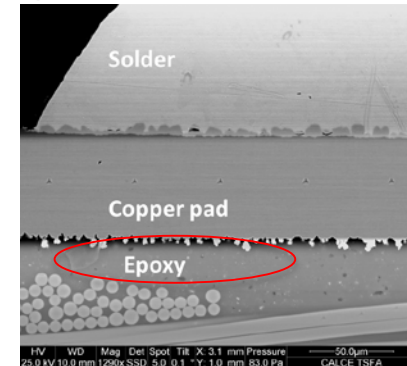
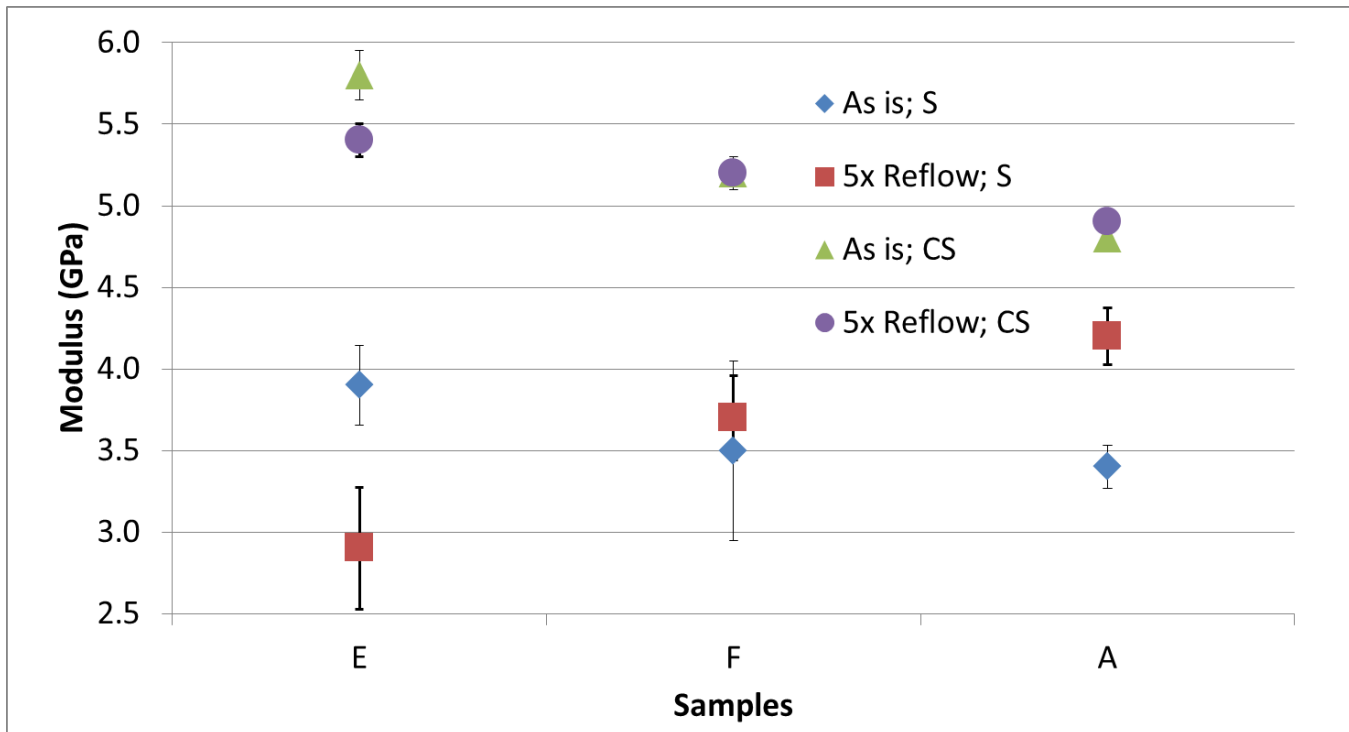
Measurement taken in
fiducial soldermask keepout



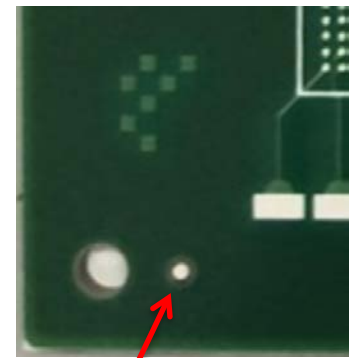
Larger measurement variation is due to surface roughness



Epoxy Moduli Comparison Between Different Locations



CS: Cross-section measurement between pad and glass

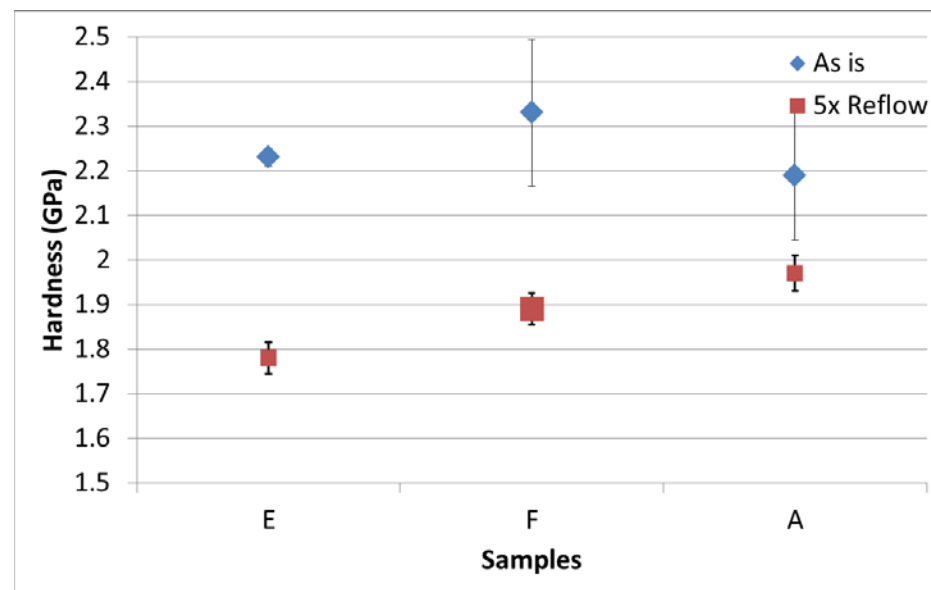
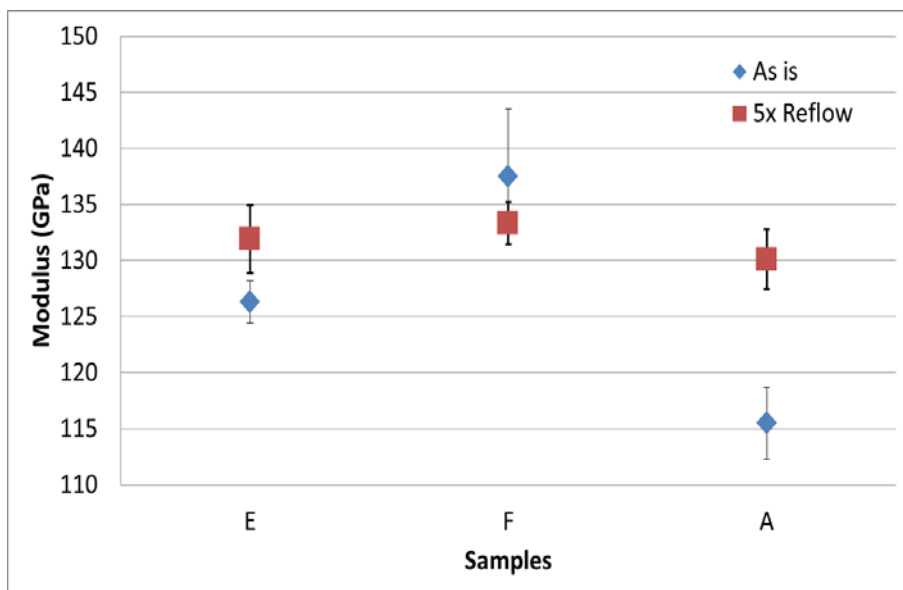
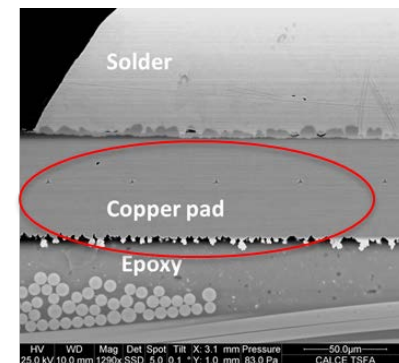


S: Surface measurement in fiducial keepout

- 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.



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-free-compatible 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.



Questions?