Examination of Common Delamination Resistance Tests for Electrical Grade Laminates

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Abstract

The delamination of electrical grade laminates continues to be a vexing problem for the printed circuit board industry. Laminates are commonly tailored to meet specific thickness and dielectric requirements. This will typically involve modification of the laminate thickness and resin content, leading to the inevitable creation of weak areas within the construction. The current industry standard for delamination testing for electrical laminates requires examination of the fracture mechanics over a mixture of mode I and mode II type behavior. The mixed mode bending leads to a good deal of ambiguity in the experimental results, complicating investigations to determine the root material properties responsible for delamination failures. To elucidate the true sources of composite delaminations, it is important to begin with an appropriate testing approach. In this paper, we examine several current and experimental delamination test methods. Methods examined include testing in pure mode I, in pure mode II, and in mixed mode I/II. Testing is performed on polymer matrix, e-glass reinforced electrical grade laminates at 23°C. From the analysis, a dedicated testing procedure is presented with the goal of more accurately predicting what values indicate a greater probability of short-term laminate failure. Based on the results, with use of laminated composite fracture mechanics, board constructions and processing conditions can be tailored to limit conditions that place unwarranted stresses on the system, thereby increasing overall laminate performance.

Introduction

Printed circuit board (PCB) delamination has been an issue in the electronics industry since the first X grade and later CEM grade materials were used for terminal boards and single sided PCB's. Advances in the polymer matrix from phenolic materials to current high end polymers, as well as advances in the laminate reinforcement from simple cotton fibers to e-glass, and in some cases quartz, have greatly increased the mechanical durability of electrical laminates, while greatly reducing the occurrence of board delaminations (1). However, the next generation of electrical laminates will be forced to withstand thermal and mechanical loads that would have been unthinkable even 20 years ago.

Movements in the industry towards greater environmental awareness and accountability have resulted in the acceptance of lead-free solders and procedures as well as halogen-free laminates, to name a few. As a result, electrical laminates are now required to withstand soldering temperatures that are on average 40oC higher than they were two decades ago; while the move towards halogen-free flame-retardants has required laminate manufactures to seek alternative approaches meet the current Underwriters Laboratory, UL, flammability requirements. This often results in the inclusion of polymers and filler materials that would otherwise not be included in the composite owing to poor or unproven physical properties. The inclusion of such materials and the application of the increased soldering/reflow temperature, greatly increases the internal stresses within the laminate, often resulting in an increased occurrence of board delaminations (2, 3).

Unfortunately, more than just changes in raw materials and in processing temperatures induce board failures. As is often the case, improper processing by the original laminator or by the subsequent board shop or OEM manufacturer will cause the composite to have undesirable mechanical properties. Common errors resulting in laminate failure include poor mixing of the polymer matrix and improper lamination temperature (2). In addition, no standard, universal test exists to quantify the stresses required to induce laminate failure.

The purpose of this paper is to examine the common delamination tests that are currently used by various laminate manufacturers and by various polymer matrix laminate industries in order to develop a more universal delamination test method.

Experimental Procedure

Three common delamination tests were examined for this experiment, namely 1. Interlaminar Bond Strength, which induces a mixed mode I/II type failure, 2. Interlaminar Shear Strength, inducing a mode II type failure, and 3. Laminate Fracture Toughness, which induces a mode I type failure. For each test method, the laminate was examined at ambient temperature, ~23°C. Several laminate lots were examined for with each test, some with possessing known good physical properties and some with intentional flaws. The purpose of the experiment was to determine if the test methods could differentiate between known good and known bad material, and to what degree of accuracy.

For the Interlaminar Bond Strength (IBS) test method, a peel tester was employed. The IBS test method is essentially a modified peel strength test method, where a board is intentionally delaminated to start and the two opposing sides are attached to opposing clamps attached to a crosshead. The crosshead sides are then separated at a given strain rate, thereby inducing a modified tensile load to the delaminated board. This tensile load acts to further delaminate the material, from which the bond strength is measured in foot-pounds, ft-lb, or force over applied width Figure 1. This bond strength value is then compared to a known good value and the material is either then passed or rejected by the appropriate quality assurance person.

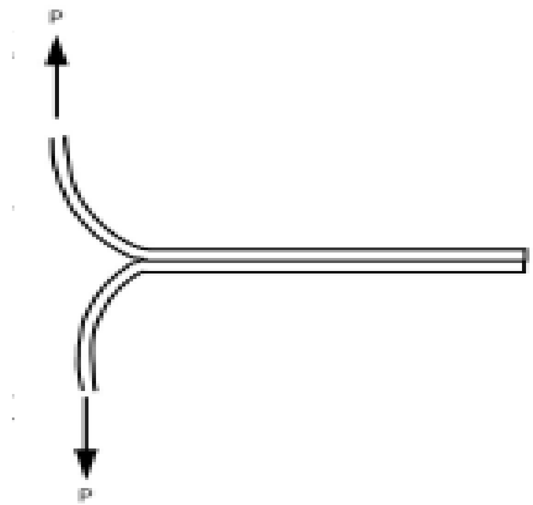
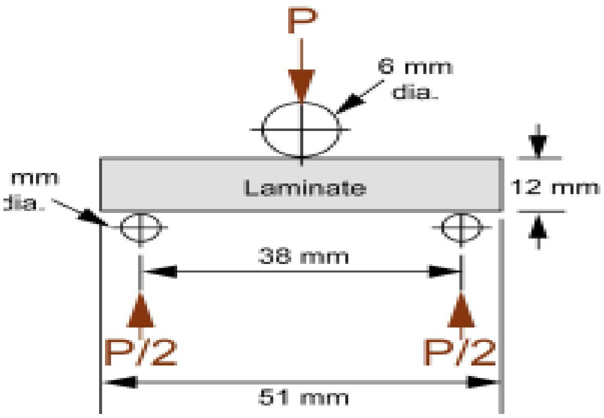


Figure 1 – The Interlaminar Bond Test Method.

For the Interlaminar Shear Strength (ILSS) test method, the ASTM-D 2344 test method was employed. The method is more commonly known as the short beam shear strength test method and essentially measured the amount of shear strength required to induce a delamination within a short laminated beam. The test method requires a laminate of approximately ¼" in thickness, t, by ½" in width, w, placed between supports 1.5" apart. A down force, P, is then applied directly between the supports, thereby inducing a delamination. This three-point bend type configuration is shown in Figure 2. The ILSS value is measured in pounds per square inch (psi) following the equation:

$$ILSS = \left(\frac{0.75 * P_{max}}{w * t} \right) \qquad (1)$$

Like the IBS test method, the values are compared to a known good and either passed or rejected. Of concern with the ILSS test method is the occurrence of "false-failures", where the laminate beam fails, but by a mode other than shear, Figure 3. Great care must be taken to ensure that all test failures are by shear. In order to do this, the sample dimensions must be kept to a tight tolerance, with the ratio between thicknesses kept to 2:1 and the length to thickness and length to width kept at 3:1 and 6:1 respectively. For the Laminate Fracture Toughness, or K_{IC}, the ASTM-D 5045 method was employed. Fracture toughness is essentially a measure of a materials resistance to cracking and crack propagation (3, 4). In the case of a laminated composite, where layer interfaces are essentially weak points, or precracked regions, the test method may be employed to determine the delamination resistance of the material.



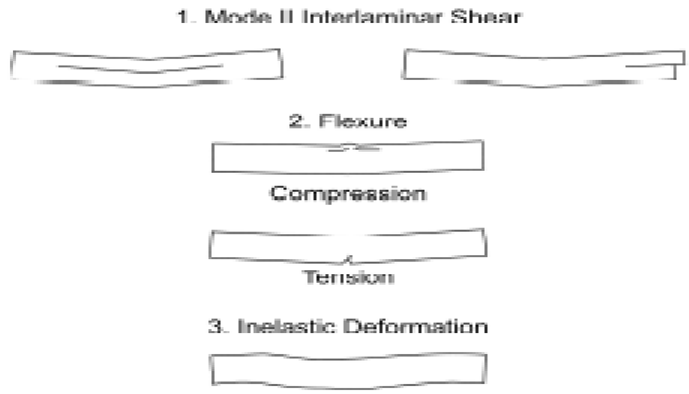


Figure 2 – ASTM-D 2344 Interlaminar Figure 3 – ILSS Modes of Failure Shear Stress Method

The K_{IC} method uses a similar test setup to the ILSS test method, with the one exception being that the supports are now placed 2” apart. Also unlike the ILSS test method, the K_{IC} test method makes use of a notch to induce material failure. The notch acts as a stress concentrator, forcing ply delaminaton at the tip, Figure 4. The notch is cut into the sample to a depth, a, of between 45% and 55% of the width, too deep or too shallow and the test method will yield invalid results. The K_{IC} value is measured in Mpa/m1/2 and is calculated from the equation:

(2)

$$K_{IC} = \left(\sqrt{P_{max} * t * w}\right) * f(x)$$

where *f(x)* equals:

$$f(x) = \frac{6 * \left(\sqrt{x}\right) * \left((1.99 * x * (1 - x) * (2.15 - 3.93 * x + 2.7 * x^2)\right)}{(1 * x) * (1 - x)^{3/2}} \quad (3)$$

where x equals:

$$x = \frac{a}{t} \quad (4)$$

In practice, *f(x)* is typically calculated based on known values of *x*, where *x* is rounded to the nearest 0.005. In this way, specific set values of *f(x)* between 0.45 and 0.55 can be used, thus avoiding long computations.

For the experiments, sets of twenty laminates of known good and known defective quality were randomly tested with each method. For each set, ten good laminates and ten defective laminates were randomly tested. The test values were calculated and compared with each other in an attempt to determine the number of defective laminates from the randomly tested lot. These values were then compared with a materials key and the success rate of each test method was determined.

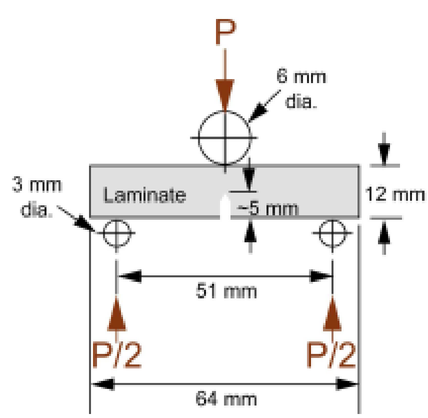


Figure 4 – ASTM-D 5045, Fracture Toughness Method

In order to quantitatively differentiate between laminates of known good or poor quality, the laminates were subjected to leadfree solder float and T-288 tests. For the lead-free solder float test, samples from each laminate were placed on a molten solder bath to induce blisters; the time to blister was measured from the initial time to the appearance of the first blister. For the T-288 testing, laminates were placed in an oven at 288$_o$C to induce blistering; the time to blister was measured from the initial time to the appearance of the first blister. Laminates with solder float values greater than 400 seconds and T-288 values greater than 15 minutes were considered to be of known good quality. Laminates with solder float values less than 60 seconds and T-288 values less than five minutes were considered to be of known bad quality.

Results and Discussion

The initial testing was performed using the IBS test method. Following the procedures laid out in the procedures, a set of twenty laminates comprised of ten each of known good and known bad board was randomly tested. The results of that experiment are plotted in Figure 5. Given the distribution of high and low quality laminates tested, a bimodal distribution of test values can be expected. Based on the results presented in Figure 5, this distribution is not evident. Comparison of the results with the laminate key suggests that the IBS test method cannot differentiate between high and low quality laminates. If, for example, we separate the data based on a typically accepted laminate threshold value of 4 lbs, then we see that nearly all of the material is accepted as having sufficient quality, with a pass rate of 85%. If this distribution is examined in terms of the mean tested value, then that success rate falls to 60%, better but still above the 50% known. Unfortunately, the test data is too closely distributed, meaning that the threshold needed to differentiate good and bad material lies within the experimental error.

A good deal of the ambiguity associated with this test method lies with the testing method itself. The method for initiating ply delaminations, that is, the insertion of a razor to start the material delamination is imprecise at best. Subtle variations in the location of the razor and the extremely low probability that the initial induced delamination lies between laminate plys means that several interfaces and induced stresses need to be accounted for to yield an accurate final value. Of additional concern is the reported final value, reported as a force rather than a stress. The test method does not have accountability to accommodate a standard delamination length. Therefore, samples that are allowed to run longer will tend to have lower values than those run for only a short time. Without this critical parameter, the test method is subject to multiple variations and errors owing to individual test operators.

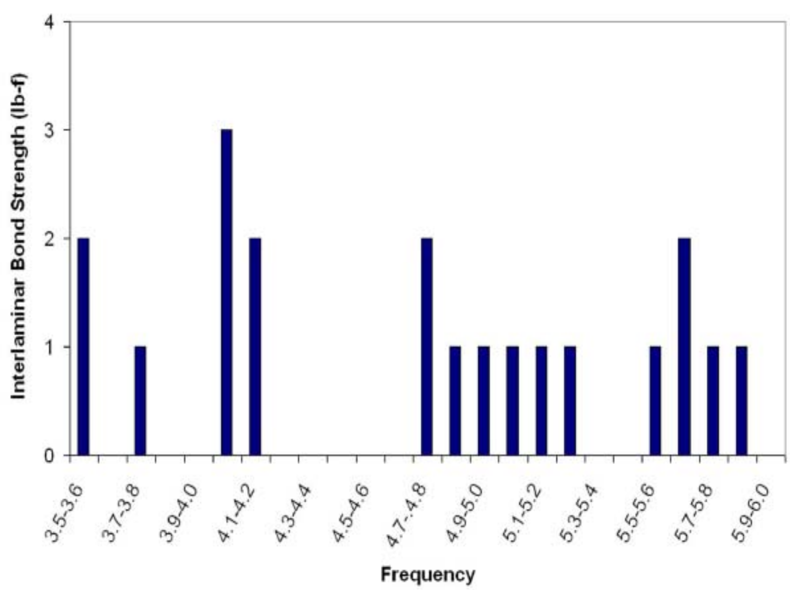


Figure 5 – Interlaminar Bond Strength Results

The second set of testing was performed using the ILSS test method. For this section of the experiment, two sets of samples were cut, one to the correct sample size and one intentionally to the incorrect width. The purpose for this intentional error set being to compare samples failing by shear and by tension.

Examination of the test results for both the correct and incorrect samples shows that both samples have bimodal distributions to various degrees, Figures 6 and 7. Taking the mean as the standard for determination of the laminate quality, there is a 45% acceptance rate for samples failing by shear, a 55% acceptance rate for samples failing by tension. When compared with the material key, the samples that failed by shear are completely correct. Since it is better to exclude good samples than to let poor quality samples pass, this lower success rate can be taken as sign that the ILSS will work as a quality control test with some calibration.

The success of the samples, especially those failing by shear can be explained in part by the general nature of the laminate. In the composite, the interlaminar interface is the weakest point in the composite. Given that the loading in the test method is applied in a direction perpendicular to the sample, if follows that the maximum stress in along the interface would be a shear force (5). If the same loading conditions are applied to all the samples, those with weaker interlaminar interfaces will fail at much lower applied loads. This differentiation between strong and weak samples is somewhat skewed for samples that do not fail by shear, owing in large part to the fact that a different failure mode apart from interfacial shear is in effect.

The final set of tests was carried out using the K_{IC} test method. Like the ILSS test method, two separate sets of samples were examined. The first set samples were examined with the proper notch sharpness and depth. The second set of samples was examined with a dull notch cut to the correct depth.

Examination of the test results again indicates a bimodal distribution in both the sharp and dull-notched samples, Figures 8 and 9. Again taking the mean as the quality threshold, the samples tested with the sharp notch have a 45% pass-fail rate, samples with a dull-notch have a 50% pass-fail rate.

This initial success taking only the mean as the threshold suggests that the distribution in the test method is truly bimodal and that the test method is a strong indicator of laminate quality that is fairly robust to variations in the test sample. The primary reason for the test methods' success lies in the fact that the method allows the material to find its own natural points of weakness. The depth and location of the notch allows for cracks emanating from the notch to stabilize and propagate along the interlaminar interface. In such a way, the laminate quality can be easily measured. Decreases in the notch sharpness causes some loss in test quality, where cracks emanating from dull notches require greater distances to stabilize, and do not do so necessarily along ply interfaces.

Based on the experimental results presented above, it is evident that the current common IBS test method is of extremely poor quality. Better methods to predict a boards' propensity towards delamination do exist in the form of the ILSS and K_{IC} test methods. Even with the mechanical testing, however, there will always be some degree of uncertainty associated with electrical laminate manufacturing. The best possible approach to quality control involves measurement of a wide range of material properties, including ILSS or K_{IC} testing, solder float times, T-288 times, as well as numerous other tests. There should never be a case where only one material property dictates material success. It is up to the laminate manufacturer, the end user, and everyone in between to determine the ultimate quality and success of a board, and the best method is to take a broad view to measuring laminate properties.

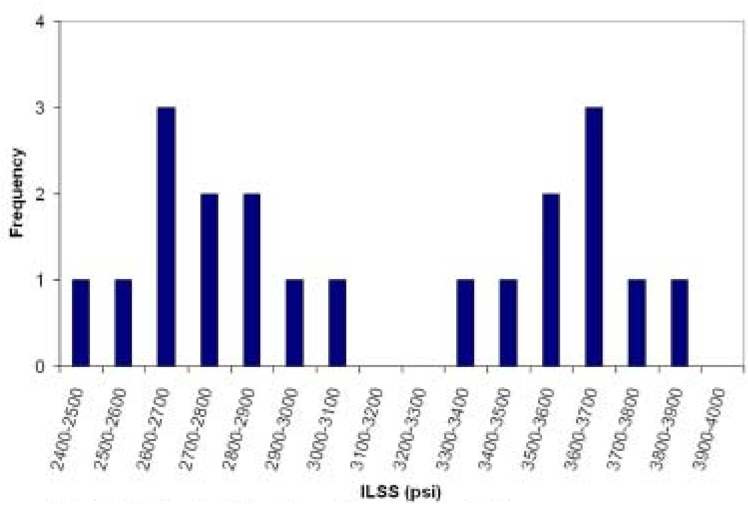


Figure 6 – Interlaminar Shear Strength Results, Shear Failures

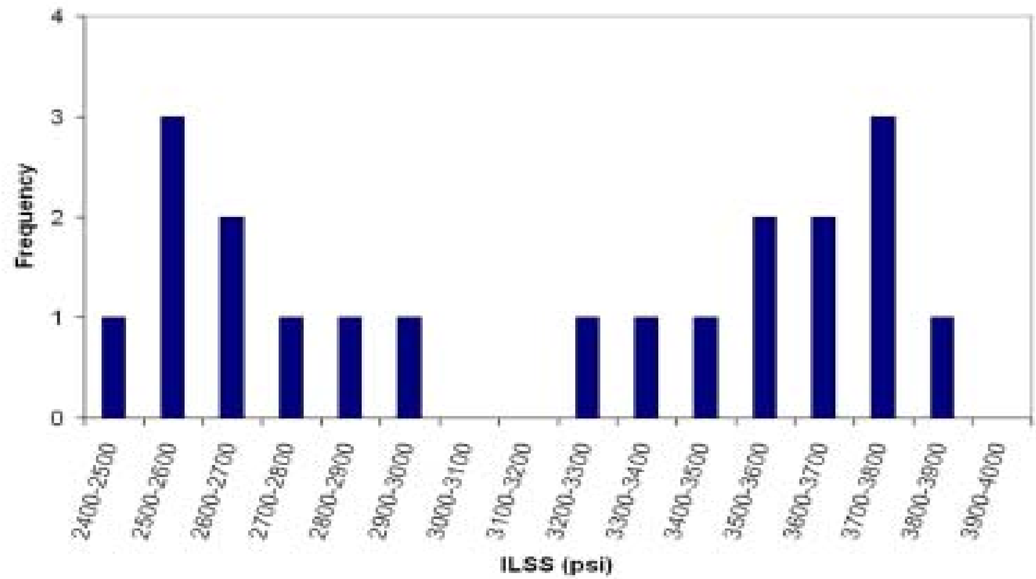


Figure 7 – Interlaminar Shear Strength Results, Tensile Failures

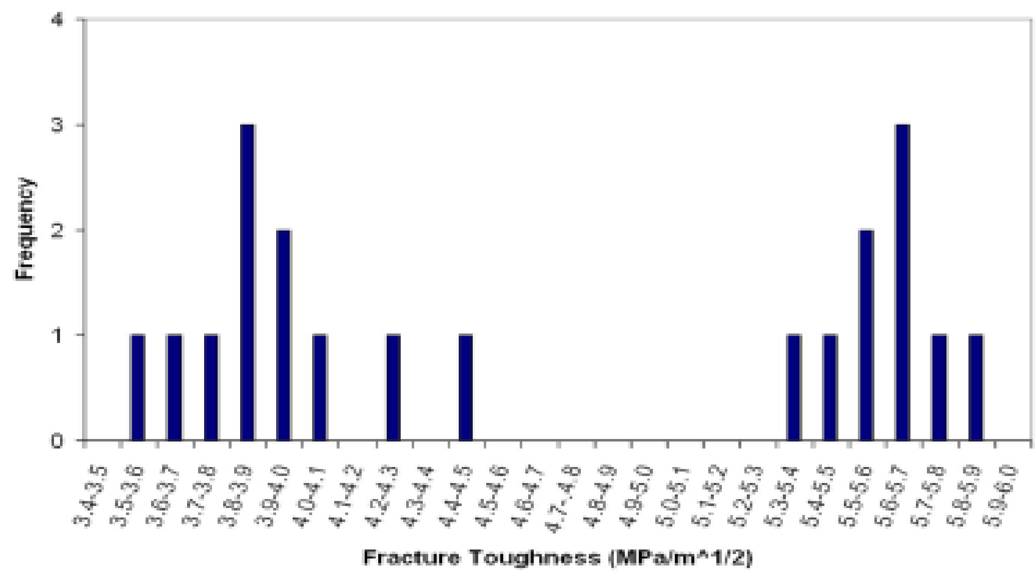


Figure 8 – Fracture Toughness Results, Sharp Notch

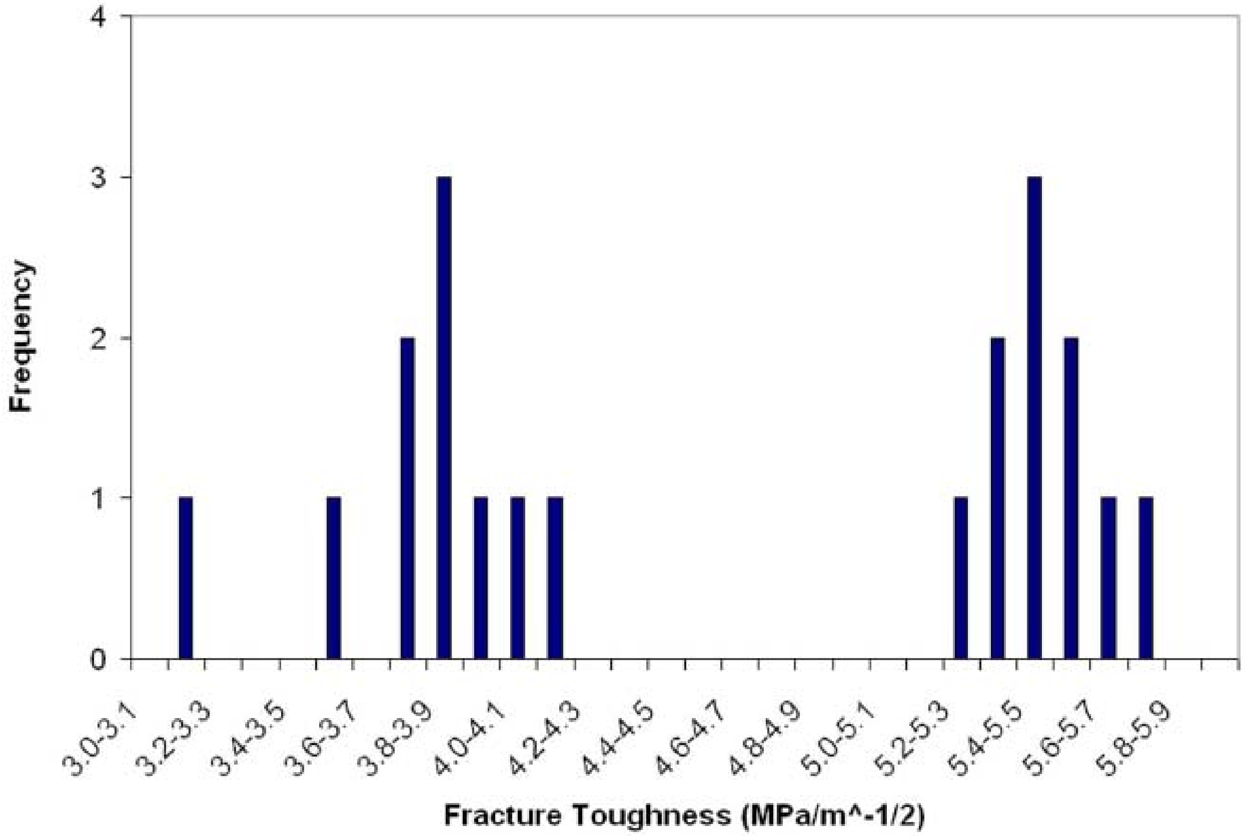


Figure 9 – Fracture Toughness Results, Dull Notch

Conclusion

Based on the experimental results, the following conclusions can be made:

- The common IBS test method does not yield accurate results when attempting to determine the delamination resistance of electrical laminates.
- The common IBS test method can be heavily influenced by both the initial test user induced delamination and by the testing machine crosshead speed.
- The ILSS test method does yield accurate results, if care is taken to ensure shear type failures.
    - For failure modes other than shear, the ILSS method has moderate success in predicting composite delamination.
- The K_{IC} test method tends to fairly time consuming but yields the best results when predicting laminate failures, given a sharply defined notch to the appropriate depth.
    - When the notch on the K_{IC} test sample is dull or cut to an incorrect depth, the test method has only moderate success in predicting board delaminations
- The best method to predict composite delamination involves testing the K_{IC} or shear ILSS in conjunction with solder-float and T-288 testing.

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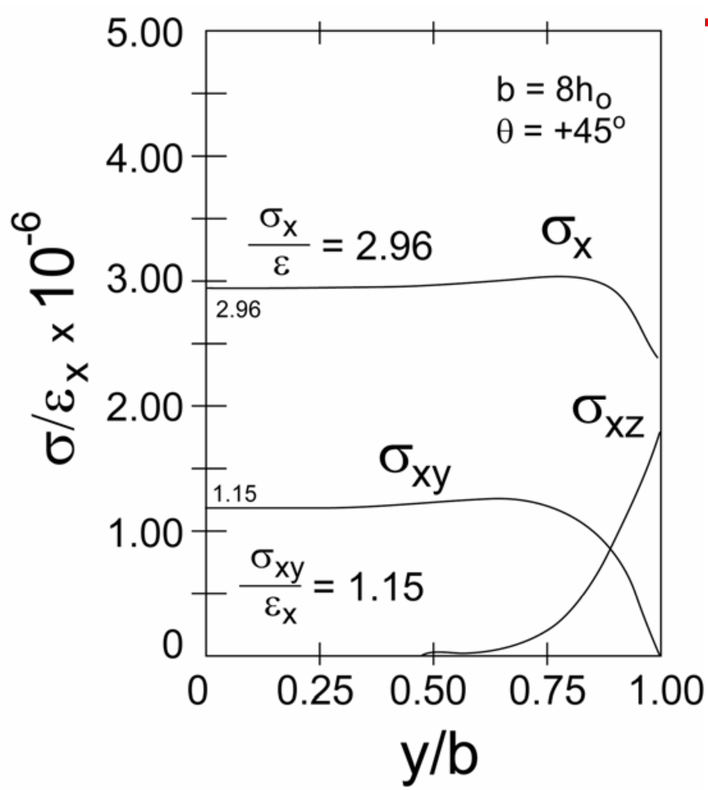
PCB Substrate Delaminations

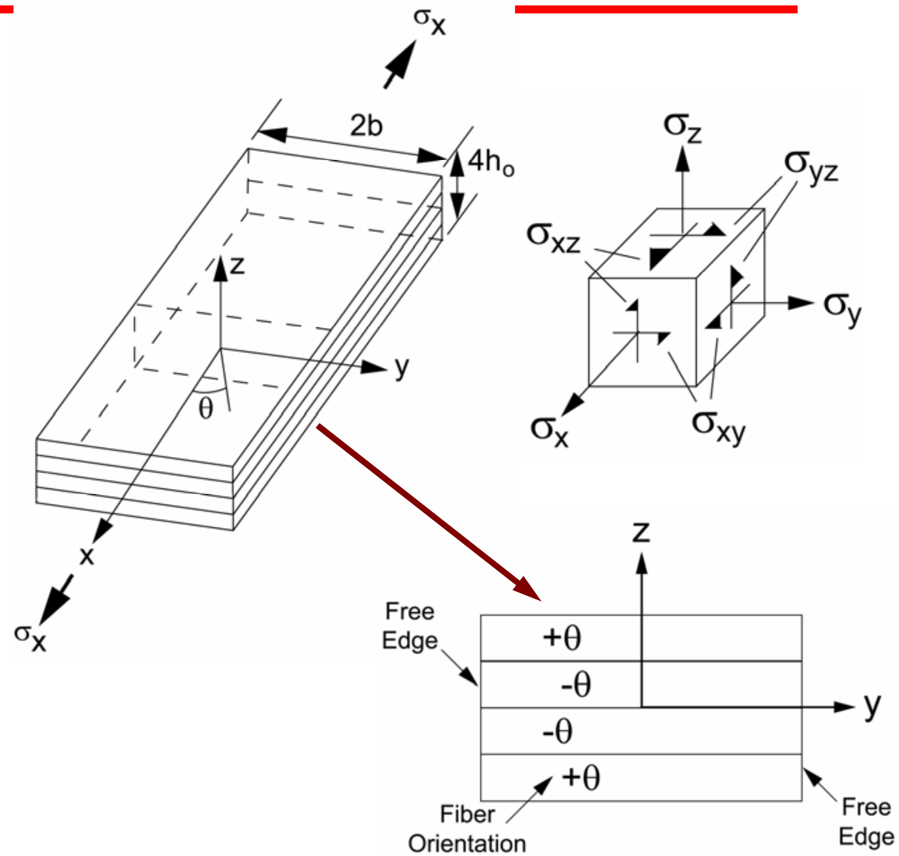
- Printed circuit boards, like all laminated composites, are subject to delamination.
- Common delamination factors include poor construction and poor processing.
- Several test methods exist to quantify the delamination strength of laminates, including:
  - Time to failure tests; solder float, T-260, T-288
  - Pure mechanical; interlaminar bond strength, shear strength, fracture toughness, flexural strength
- No universal test method, quantification procedure exists for electrical laminates.

Sources of Substrate Delaminations

- Delamination in laminated composites involves a deformation in the unreinforced direction, either uniaxially or through shear.
- Classical lamination theory assumes an infinite laminate, and that the composite exists in a state of plane stress.
- In reality, there is a transition from generalized plane stress at the free edges to a three-dimensional state of stress, thereby leading to delaminations and laminate failure.
- Work performed by Pipes and Pagano (1970), and later Pipes and Daniel (1971) confirms this transition in stress states.

Sources of Substrate Delaminations





- In an symmetric unidirectional laminate, a condition of plane stress exists at the core, transitioning to plane strain at the edges and inducing a shear strain in the x-z and y-z directions.

Pagano, Pipes, Composite Mater.,**4**, (1971)

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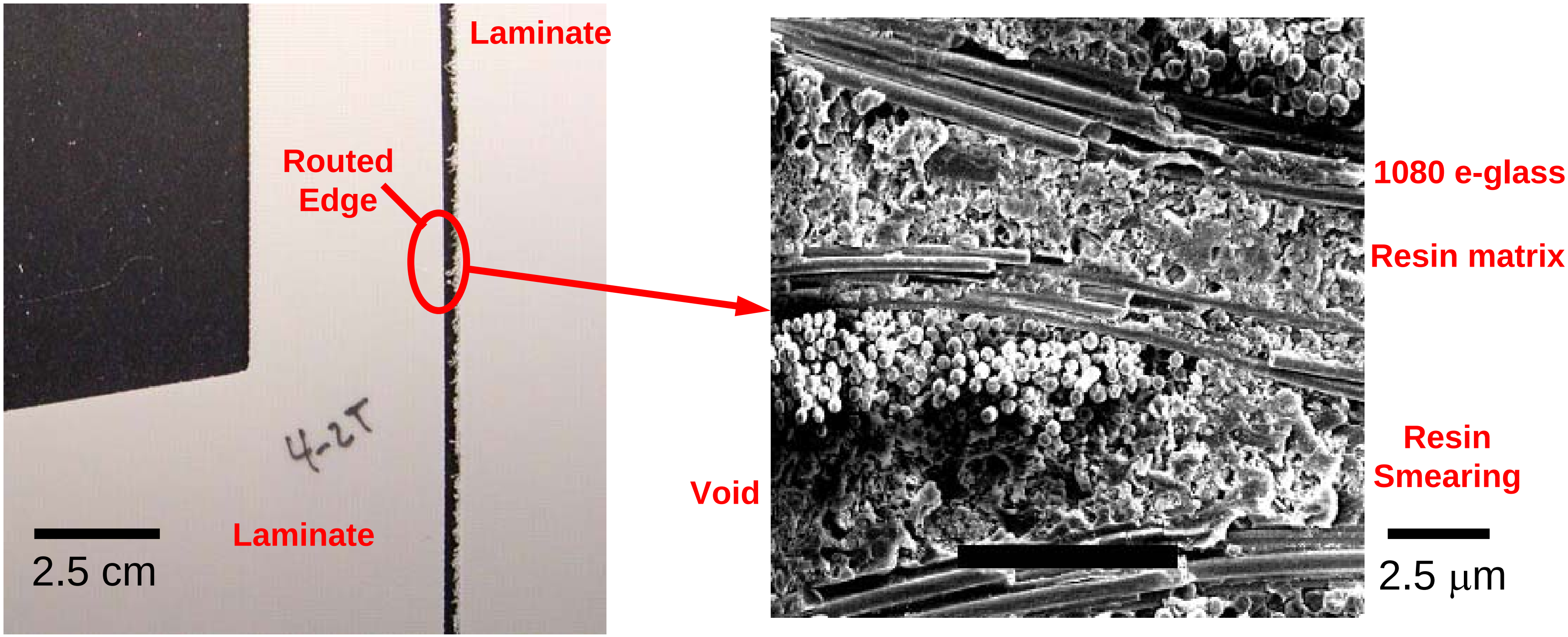
Processing Damage and Delaminations

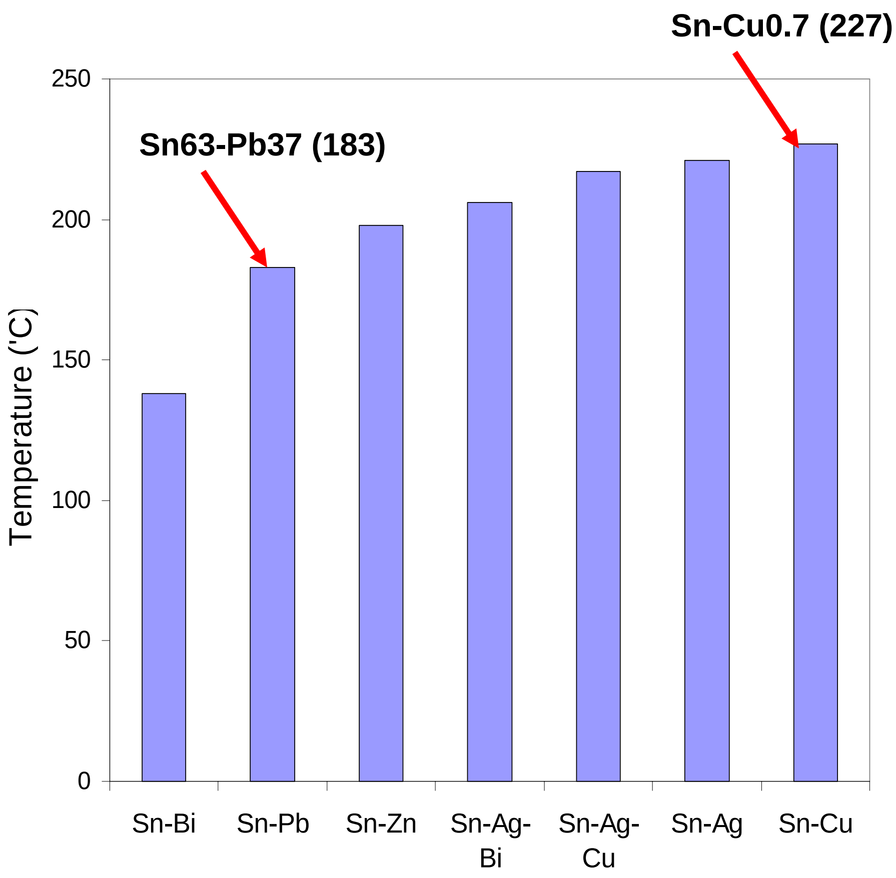


- Damage and delaminations are often induced by post lamination procedures, such as drilling and routing.
- Since delaminations result from poor interlaminar strength, it is important to be able accurately quantify this strength.

Temperature Induced Delaminations

- In the past 20 years several significant industry wide initiatives have passed including:
  - RoHS
  - Lead-free processing
  - Halogen-free processing
  - General trends towards higher T_g, T_d, lower Dk/Df

- To comply with initiatives, some less common or unproven materials need to used.
  - Thus, robust materials testing methods are required.



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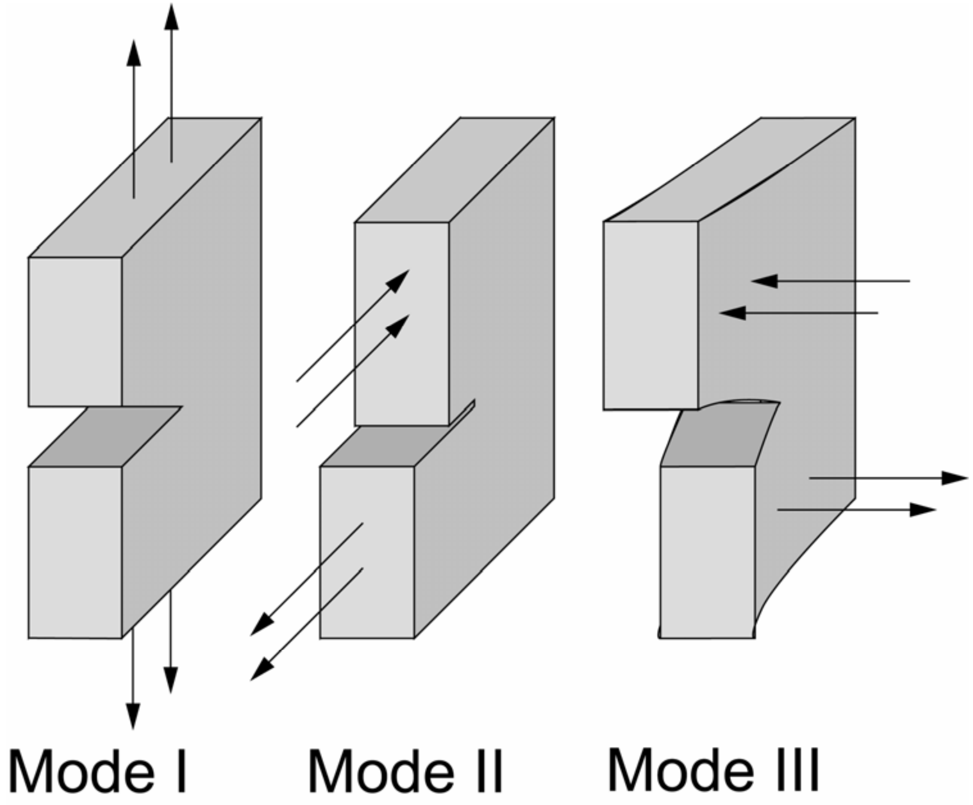
Experimental Procedure

- Depending on the stress field in the vicinity of the crack tip, three principle fracture modes are possible: Mode I, Mode II, and Mode III.

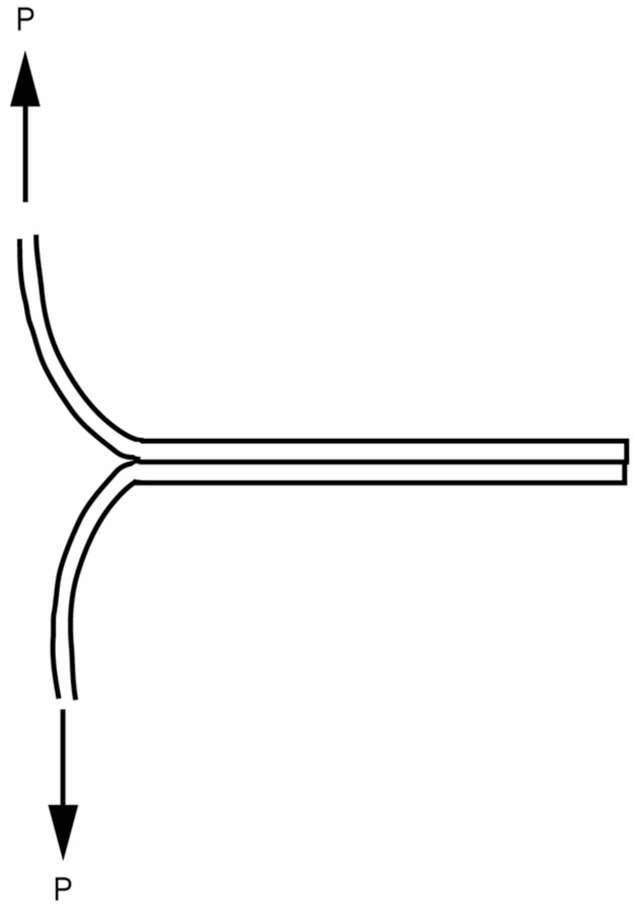
- For this experiment, three test methods were compared to determine the best indicator of delamination quality:
  - Mixed mode I/II Interlaminar Bond Strength (IBS)
  - Mode II Interlaminar Shear Strength (ILSS)
  - Mode I Fracture Toughness (K_{IC})



Interlaminar Bond Strength Testing

- The IBS test method is essentially a modified peel test.
- Intentional delamination is induced via a razor blade, separated sides are peeled apart via a crosshead
- IBS is quantified as force over length:

$$IBS = \left(\frac{Force}{Length} \right) = \left(\frac{P}{L} \right)$$



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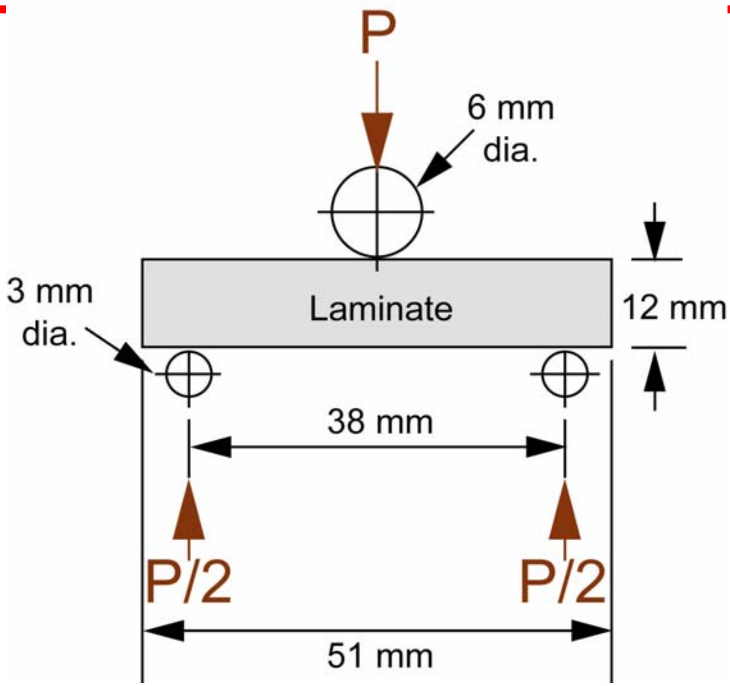
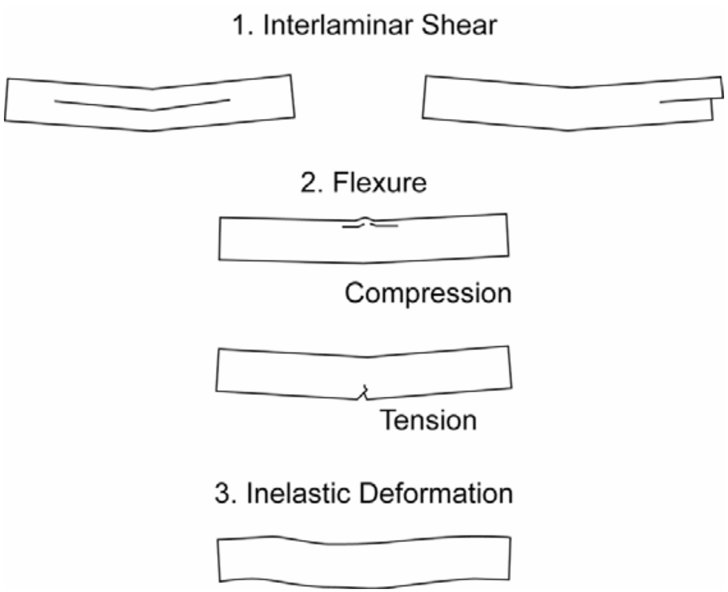
Interlaminar Shear Strength Testing





$$ILSS = 0.75 \cdot \frac{P_{\max}}{w \cdot t}$$

- ASTM-D2344 is a simple three-point bend of an exceptionally thick laminate to induce inter-ply failures.
- The increased thickness is to ensure shear failures, rather than flexural.

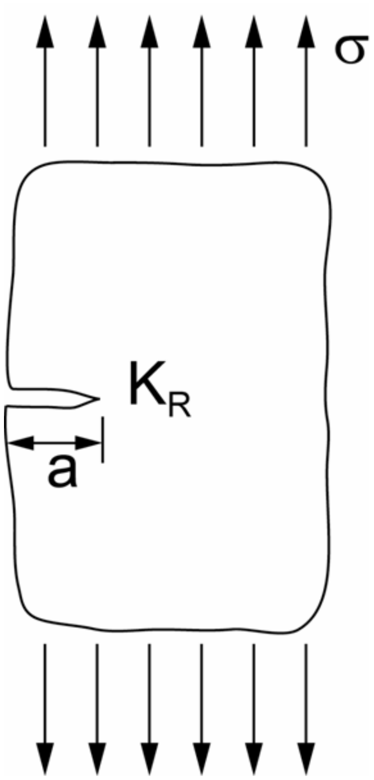
ILSS & K_{IC} Testing

- The ILSS was measured using ASTM-D2344 on an electric drive load frame.
- The fracture toughness, K_{IC}, was tested using ASTM-D5045.
- The K_{IC} and ILSS methods use similar geometries and loading conditions.
  - The difference being K_{IC} samples are notched to induce Mode-I failures.

Fracture Toughness, K_R

- Given a defect acting as a stress concentrator in a material of a given length *a*, there exists a stress intensity factor, K, that can be measured to gauge the impact of the defect.

- With any given material, there exists a value of K that denotes the value below which the defect will not propagate a crack, denoted as K_R.



Measurement of K_I and K_{IC}

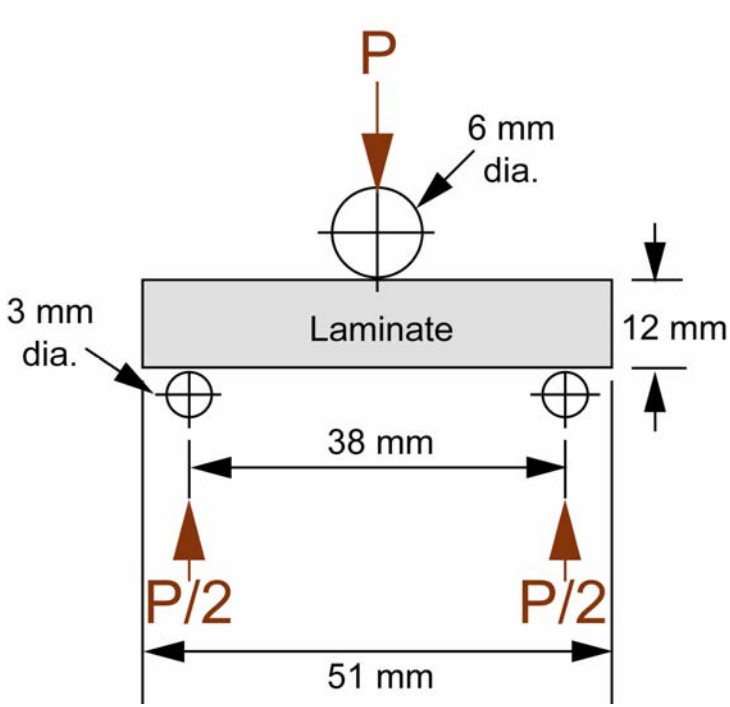
- K_I, as a measure of Mode I crack resistance, is a function of the Mode I applied stress, the defect size, and the defect geometry following:

$$K_I = Y\sigma\sqrt{(\pi a)}$$

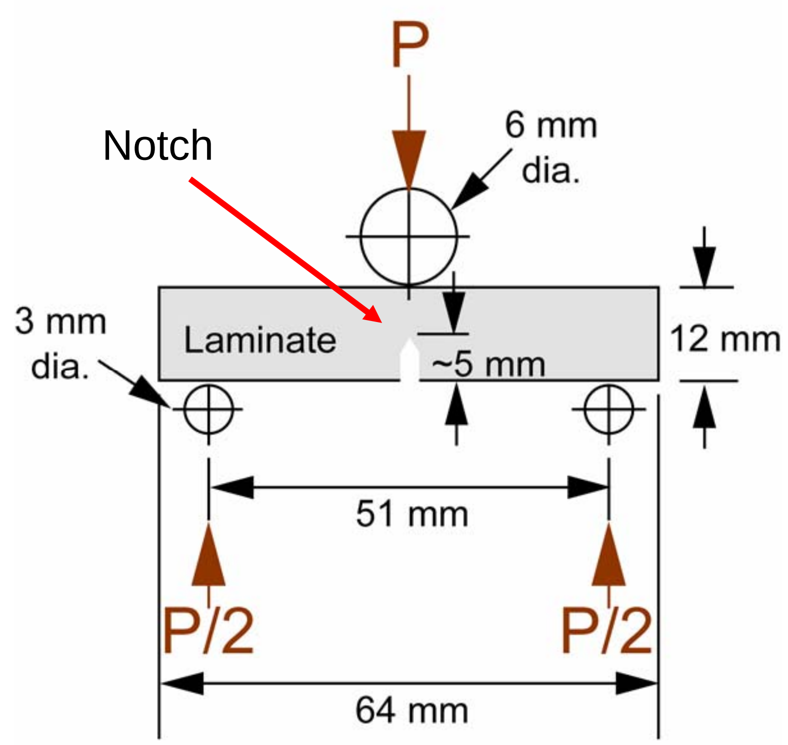
  - where *a* is the defect size, σ is the applied stress, and *Y* has a typical value of ~1.12 for small cracks depending on geometry and location

- K_{IC} is an inherent material property measuring the material resistance to an impinging crack, independent of material thickness, *t*.
  - In order to measure K_{IC} most testing protocols call for single edge notch sample with an *a/t* of between 0.45 and 0.55.

K_{IC} vs. ILSS



$$ILSS = 0.75 \cdot \frac{P_{max}}{w \cdot t}$$



$$K_{IC} = \left(\sqrt{P_{max} * t * w}\right) * f(x)$$

Where *f*(x) is a function of x and:

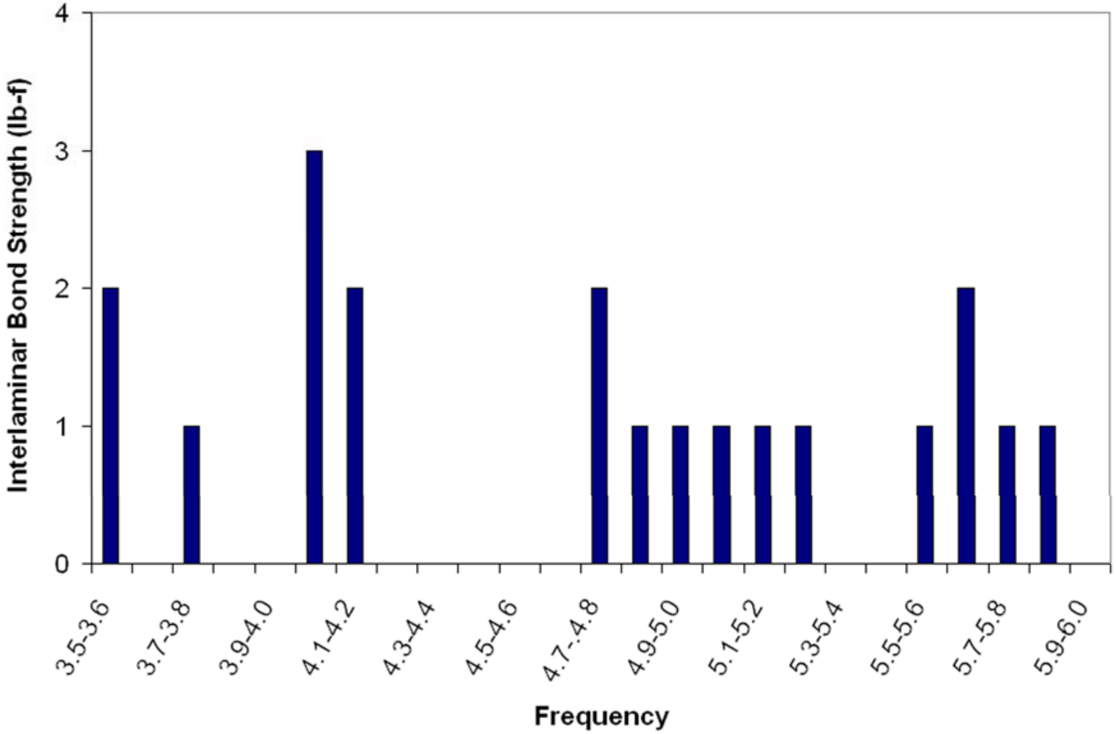
$$x = \frac{a}{t}$$

Experimental Procedure

- For each of the three testing procedures 20 samples were examined, 10 of known good quality and 10 with known poor quality
  - Good" quality samples possessed solder float test values greater than 400 seconds and T-288 (IPC 2.4.24.1) values in excess of 15 minutes.
  - "Poor" quality samples possessed solder float test values less than 60 seconds and T-288 values less than five minutes.

- For the ILSS and K_{IC} tests additional factors were examined, including:
  - Thickness of the ILSS samples to induce both shear and tensile type failures
  - Notch geometry of the K_{IC} samples to affect the crack growth behavior and fracture toughness.

Interlaminar Bond Strength



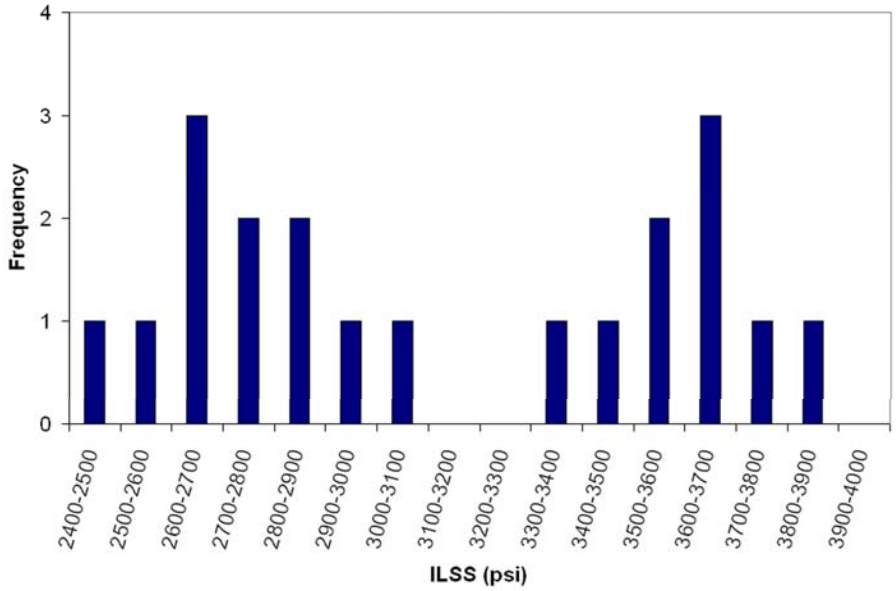
- Using a threshold of 4-lbs, nearly 85% of samples are accepted as having sufficient quality.

- Using the mean IBS value, the pass rate falls to 60%, meaning 20% of "poor" quality samples are accepted.

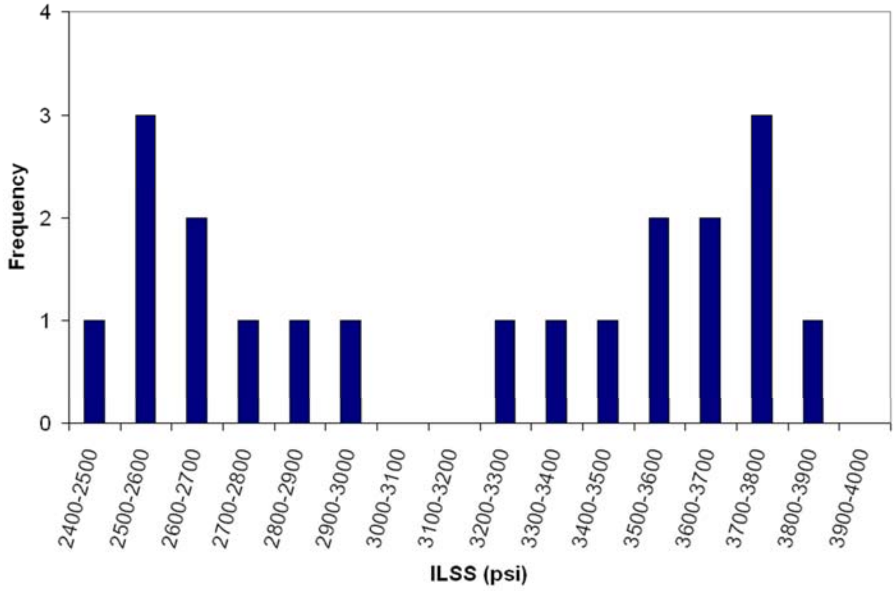
Interlaminar Bond Strength

- Given the large disparity in known sample quality, a bimodal distribution of the test results can be expected.
- Using 4-lbs as a pass value, nearly all “poor” quality samples are allowed to pass.
  - Tightening the quality spec to the mean still allows “poor” quality samples to pass.
- Use of razor to start delamination introduces operator error,
  - Subtle variations in the location of the initial cut will alter stress state.
  - Low probability that the initial delamination is limited to just two plys.
- Final value reported as imprecise force over length, not a stress.
  - Method cannot account for different delamination lengths, longer run samples will have lower values.

Interlaminar Shear Strength



Shear Failures, 45% pass rate



Tensile Failures, 55% pass rate

- Two sets of samples were cut, one to the correct dimensions and one to an incorrect width to induce tensile failure.
- Results from both sample sets indicate bimodal distributions.

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Interlaminar Shear Strength

- Using the mean test value as the quality spec, samples failing by shear have a 45% pass rate, tension failures have a 55% pass rate.
  - Comparison with the sample key indicated that all “poor” quality samples are caught by shear failures, all but one are caught by tensile failures.

- It is better to exclude good samples than pass bad, the 45% pass rate is an indicator that the ILSS method will work as a quality control test.

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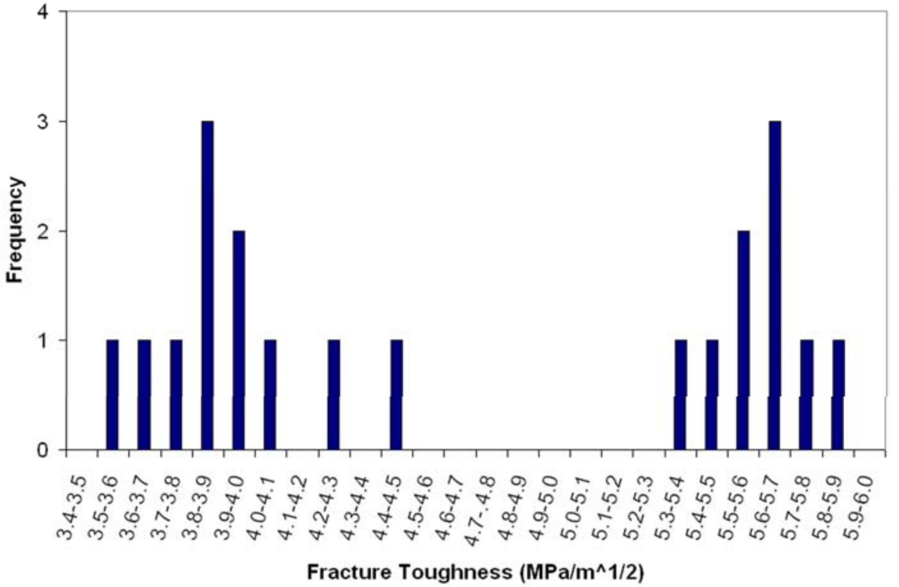
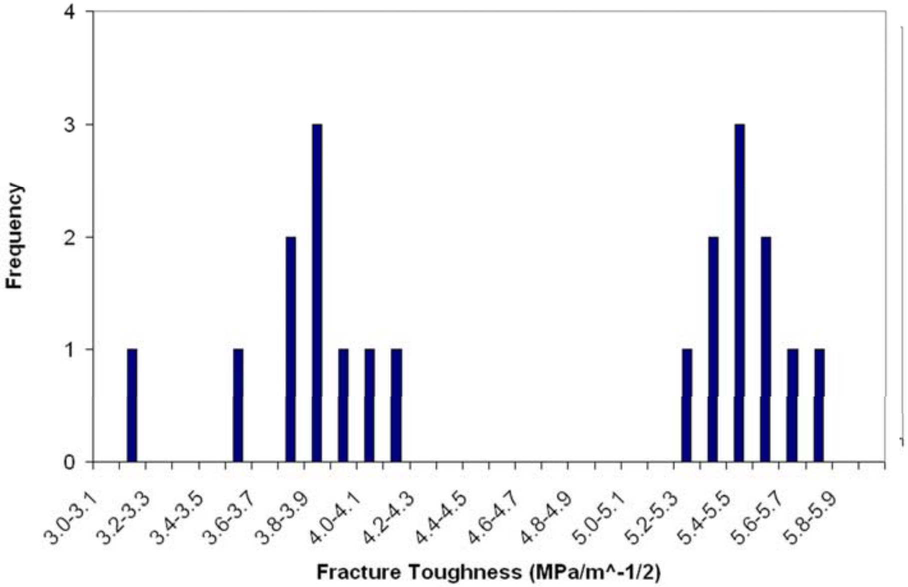
Fracture Toughness, K_{IC}



Sharp Notch, 45% pass rate



Dull Notch, 50% pass rate

- Like ILSS, two conditions were examined, one with a sharp notch and one with a dull notch.
  - Both conditions used notches cut to a depth of 0.45*t
- Examination of the test results for both conditions indicates bimodal distributions exist.

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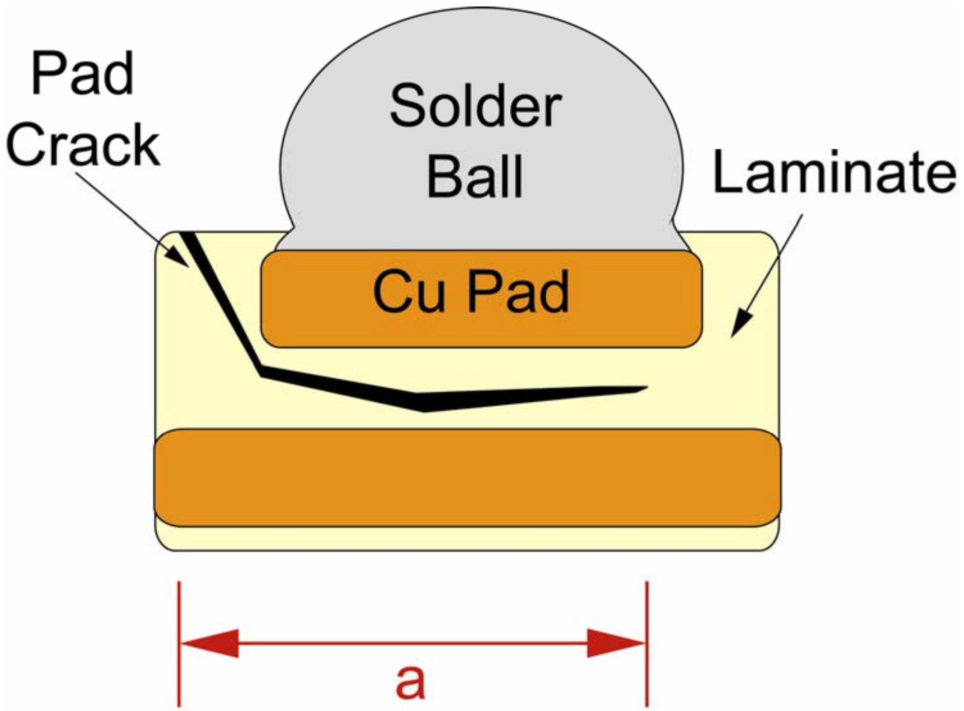
Fracture Toughness, K_{IC}

- The notch geometry impacts the stress state at the start of onset of “cracking”, in this case “cracks” being delaminations.

- The K_{IC} test relies on cracks from the notch finding stable propagation paths.
  - Samples with a dull notch can be expected to take longer to initiate and to stabilize on an interply plane.

- In practice, K_{IC} can be extremely time consuming, likely limiting use of the test.

K_{IC} as a measure of Pad Cratering, CAF



- True laminate cracks are commonly seen in conjunction with pad cratering and with CAF type failures.
- Quantification of the laminate K_{IC} could yield information regarding the applicability of specific systems to specific applications

Conclusions

- The IBS test method appears to be a extremely poor measure of laminate quality.
- ILSS and K_{IC} test methods appear to be good devices to measure laminate quality.
- In practice, the K_{IC} test method can prove to be time consuming, making it a less desirable quality control test.
  - K_{IC} could provide a good measure of CAF and pad cratering susceptibility
- The best approach to measure quality lies with examining a wide range of laminate properties including, but not limited to mechanical test results.

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