Peel Strength of Deposited Adhesiveless FCCL, or, Why Don't They Ever Say, "It Sticks Too Good?"

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Abstract

The peel test will be reviewed, with special attention given to deposited adhesiveless copperclads. Details of the specification are reviewed The relevance of the title subject will be addressed from the perspective that a vendor and a customer of a flexible material can head off disaster if they spend time communicating on what the requirements of that substrate material are.

We will consider the mechanics of the test, IPC specifications, will be reviewed with specific case histories in which the choice of peel method was critical to problem resolution.

We will discuss the many influences on peel strength values by presenting data on variables such as conductor thickness, conductor width, and copper treatment, as well as more subtle things such as surface finish and even simple choice of test method. Details such as the effect of surface finish will receive comment. Data concerning ENIG-plating will serve as the backdrop for this segment.

Audience members will be encouraged to participate by asking the "expert" to answer questions such as, "which test is most important," and "how much peel strength is enough?" This will be used as a teaching opportunity to exemplify the value of close communication between customer and supplier.

Failure modes will be described for deposited clad flexible substrates. The value of investigation of this characteristic will be stressed, together with other investigational techniques for the engineer who may be new to the industry. Among these nuggets will be the exhortation to learn to write good English, which will permit a partial answer to the question posed in the title.

Choice of substrate material with respect to the tiecoat will be clarified by discussion of processing and product characteristics.

A brief mention will be made of alternative methods of adhesion measurement, including shear testing and tensile testing. Again, with the author's own data on adhesiveless FCCLs, a picture will be provided of the relative strengths and weaknesses of each method.

Introduction

The use of adhesiveless Flexible Copper Clad Laminates (FCCLs) continues to grow within the industry. These systems are more chemically resistant than are their adhesive-based counterparts, and more resistant to thermo-oxidative conditions and to the effects of humidity. Superior electrical properties and reduced thickness and weight make adhesiveless substrates an attractive alternative to conventional 3-layer FCCLs.

Adhesiveless FCCLs are produced in a number of ways. Polyimide precursor can be coated on copper foil and cured, yielding "cast" products. A thermoplastic polyimide coating can be applied to PI film and foil laminated thereto, producing a "laminated" FCCL. Finally, vacuum deposition techniques can be employed to fabricate copperclad polyimide substrates. A general approach for this style of product is to sputter a tiecoat layer of a few nanometers thickness, followed by deposition of a few hundred nanometers of copper. Electroplating provides the final thickness of copper. It is the latter style of product that is the subject of this paper.[1]

Deposited adhesiveless clads are preferred for tight conductor geometry applications such as chip-on-flex, where 40µm pitch is being produced in volume. This preference arises principally due to the ease of producing the thin copper layers, as contrasted with foil-based products. Semi-additive processing becomes attractive with an economical FCCL of 2µm copper thickness.

As well, the deposited approach to adhesiveless clads enables an MTH, or Metalized Through-Hole approach to PTHs, as illustrated in figure 1, avoiding the need for electroless copper plating.

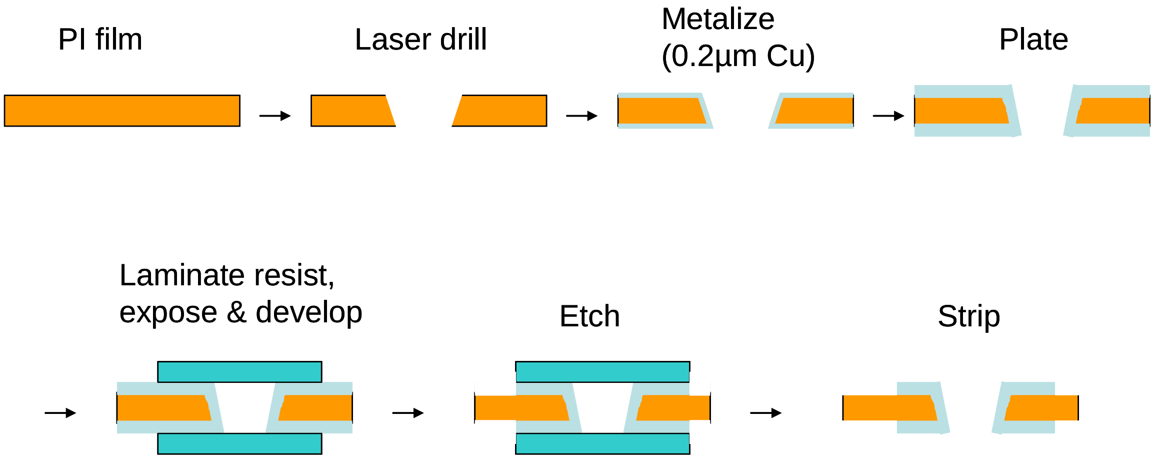


Figure 1

So, why a paper on peel testing of adhesiveless FCCLs?

Historically adhesive-based FCCLs have been built with copper foil, of course. The manufacture of the foil and production of FPC are grounded in many decades of experience, well-developed processes and thoroughly characterized control limits on such things as thickness, bulk resistivity, and treatment profile. A good baseline for the interpretation of peel strength measured on such foil-based materials is ingrained in the industry.

Deposited adhesiveless FCCLs expand the playing field considerably. Thickness down to 2µm copper is a standard product offering. The product is available with the submicron vacuum-deposited metal coating as well.

Industry specifications for adhesion measurement were developed around the limited copper thicknesses sold by foil manufacturers. Without a good understanding of the factors coming into play in the peel test, erroneous conclusions could be drawn when comparing test results. It is the objective of this paper to review some of these influential factors.

Literature/theory

Much has been written about the mechanics of the peel test.[2-6] It is not the intent of this paper to discuss theoretical foundations of the peel test. Practical observations derived empirically from application of the industry-accepted methods will be shared, with the goal of providing an awareness of constraints appropriate to generalization of peel results.

Adhesive-based materials tend to fail by elongation and then rupture of the adhesive layer. In contrast, Deposited adhesiveless clads fail by crack propagation within the polyimide matrix.

Initiation vs. propagation

Peel strength is a test of propagation of debonding force, and not one of initiation. If a particular material produces only tearing of the substrate when subjected to the peel test, one should consider how closely the specimen configuration resembles the interconnect to be produced from the material. Often, singlesided clads break the film substrate (tear) when tested as diecut specimens, but yield a completely different failure mode when narrow traces are imaged and peeled. That is to say that the reported ‘peel’ results may not be relevant to the customer’s application. One should be cautious of an assertion that “the film breaks: you can’t do better than that.” While this may be true with the test configuration employed in QC testing, one is advised to confirm that the identical failure mode is observed on the interconnect before accepting this result.

Industry specifications for the Peel test

The IPC specifies the peel test in IPC TM-650 2.4.9, and JIS standard C6471 discusses the peel test in paragraph 8. Let us look at the specifics. JIS describes two methods, differing essentially only in the angle of peel. The recommended method A is for a 90° angle peel test with crosshead speed of 50mm/min. Method B is performed at 180°, with the suggestion that it be used for films <25µm in thickness, or when it is difficult to maintain the 90° angle of peel due to tenting, or when it is “desired to obtain approximate measured values quickly.”

The test regime calls for testing a) as plated and imaged, b) after a predried 5-second 260°C solder float, and c) after a one-hour 180°C heat-aging step. Procedures for performing the test after specimens are immersed for 5 minutes in a chemical solution (2N acid, 2N base, and isopropanol are the three chemicals individually tested.) 3mm conductors are imaged for the test.

Most of us are familiar with the details of the IPC peel test. Like JIS C6471 method A, conductors are peeled at 90° angle at 50.8 mm/min (2 inch/min.). Conditioning procedures after imaging call for a) a five-second 288°C solder float and b) five cycles of -50°C to 150°C thermal cycling.

There are two significant differences between JIS C6471 and IPC TM-650 2.4.9 that deserve comment.

The first is that double-sided FCCLs are to be tested with copper etched off the backside per C6471. In contrast, the note at the end of paragraph 6.2 in IPC TM-650 2.4.9 states that it is permissible to leave the backside copper cladding in place to prevent tenting from occurring in the peel test.

The second difference is that the IPC method permits peel coupons to be prepared either by etching conductors 3.2mm (0.125") wide, very similar to JIS C6471, or by die-cutting ½" wide samples. In fact, the current revision of 2.4.9 says that the diecut method shall be the referee method. The relevant slash sheets for adhesiveless clads, 4204/11 and 4204/18, specify values for each style of specimen "As received," but only for the diecut method after conditioning

We will return to these facts later.

Factors that influence peel strength

Focusing on the deposited products, the biggest influence on peel strength is the thickness of the copper being debonded. Why is this so? Research has demonstrated that 70-90$^+$% of the force measured in a peel test goes into bending the copper conductor.[7-8] Other factors can influence this result: it has been shown that, for a polyimide-coated wafer, adhesion of sputtered copper goes ***down*** as a function of metal thickness.[8] This has been explained in terms of the degree of plastic deformation due to stiffness of the backing.

The intrinsic adhesion of the copper deposition process will influence the magnitude of contribution due to the bending of the conductor. Figure 2 shows this dependence of peel on copper thickness for two different deposited adhesiveless clads built on 25µm polyimide.

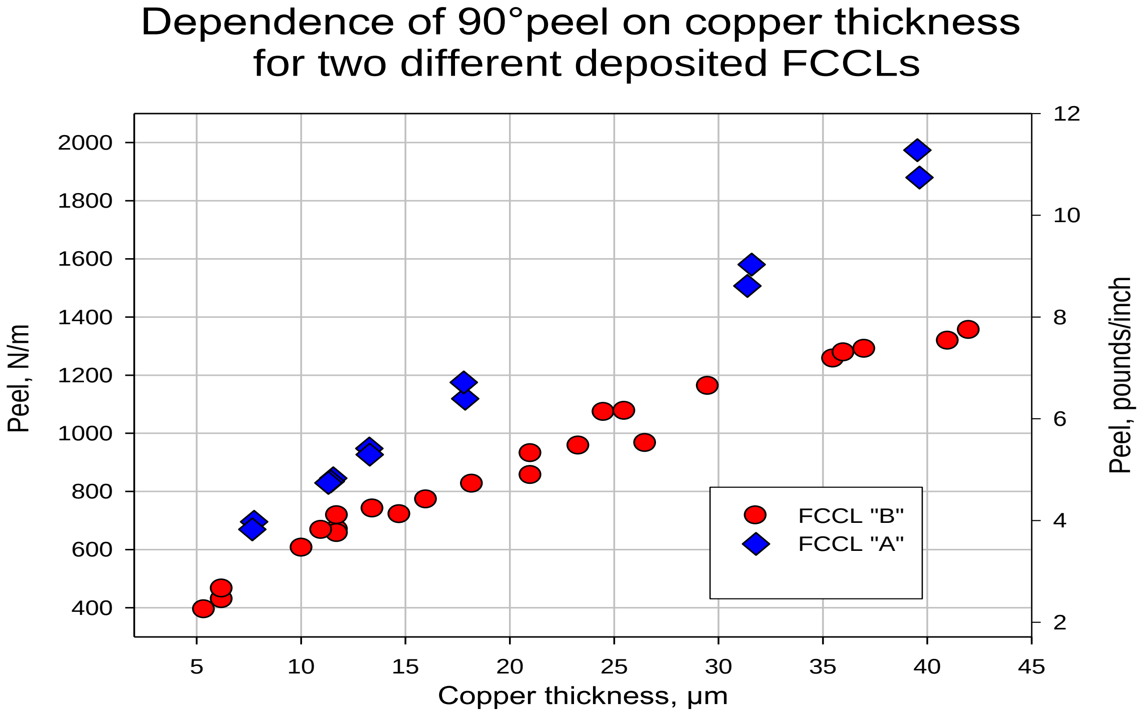


Figure 2

Chen et. al. described a theoretical foundation for the increasing slope observed for the higher adhesion product.[7]

As mentioned, angle of peel can have an effect on peel strength, but not always. Figure 3 shows the relationship between 90° and 180° peels for one particular product.

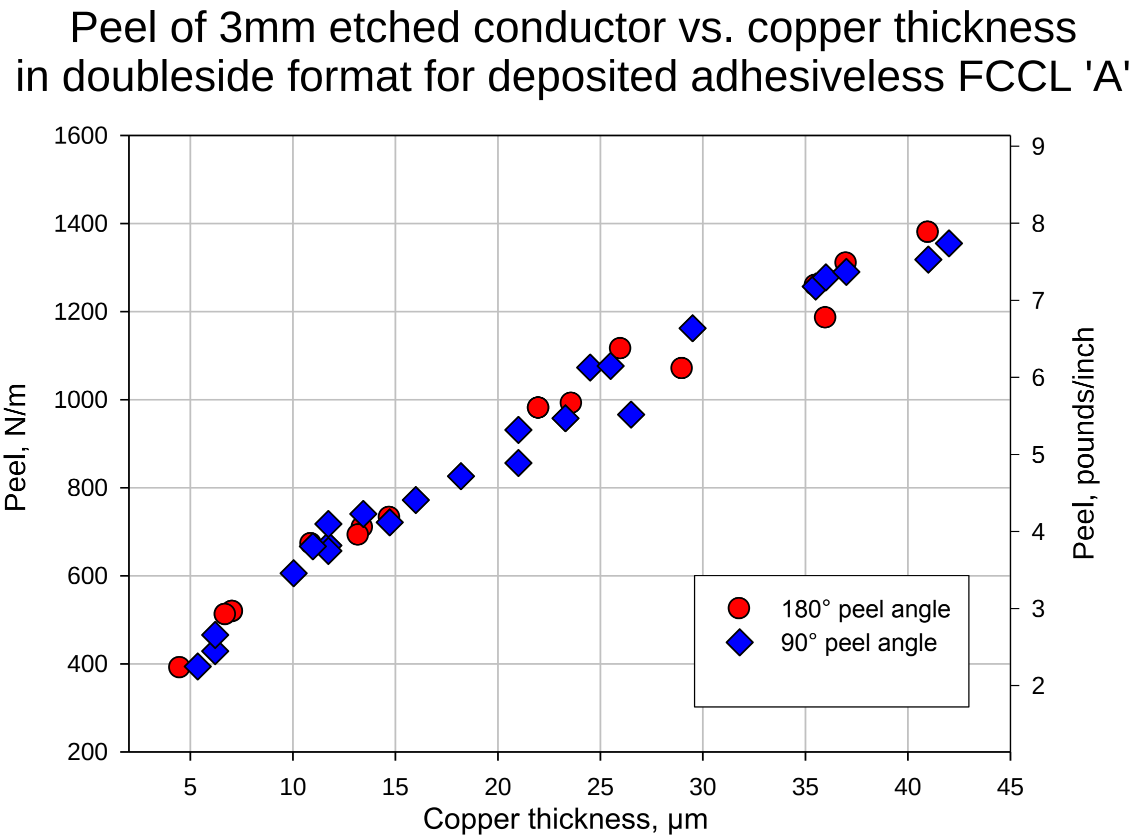


Figure 3

For a deposited product with higher overall peel strength, the picture is somewhat different. Figure 4 illustrates this data set.

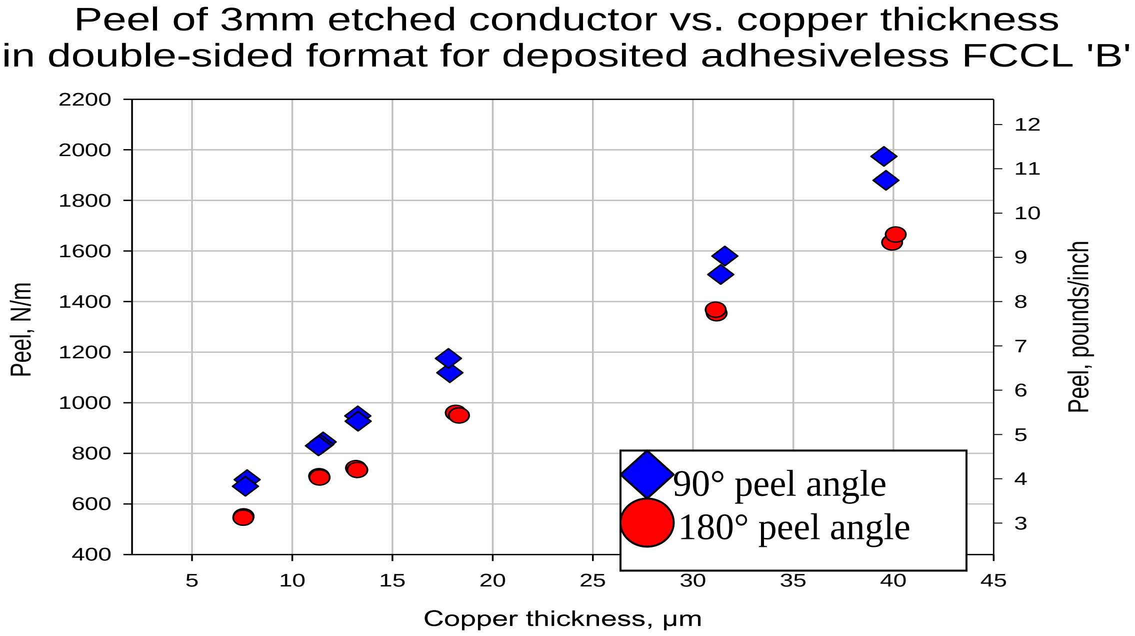


Figure 4

The difference in results obtained from these two materials indicates that care should be exercised when comparing results obtained by JIS C6471method A and method B.

Measuring copper thickness

Given that peel strength depends so obviously on the measured copper thickness, a couple of factors that influence that thickness measurement deserve mention. It is generally recognized that the area of the specimen needs to be chosen to reflect the type of feature measured. What is sometimes overlooked is that the resistivity of copper deposited by a commercial plating bath often varies from that measured on ED copper foil. Foil thickness is generally inferred from a measured resistance. That means that the standards used to calibrate should be prepared from the same type of plate-up being measured. The typical foil-on-FR4 standards will give erroneous readings when used to measure an electroplated deposited FCCL. In figure 5, crossweb thickness readings by a vendor and a customer on a single sample are displayed. Measurements were performed with the same style of resistivity measurement tool. Each side described the dip in thickness accurately, but the offset arose from a difference in calibration standards.

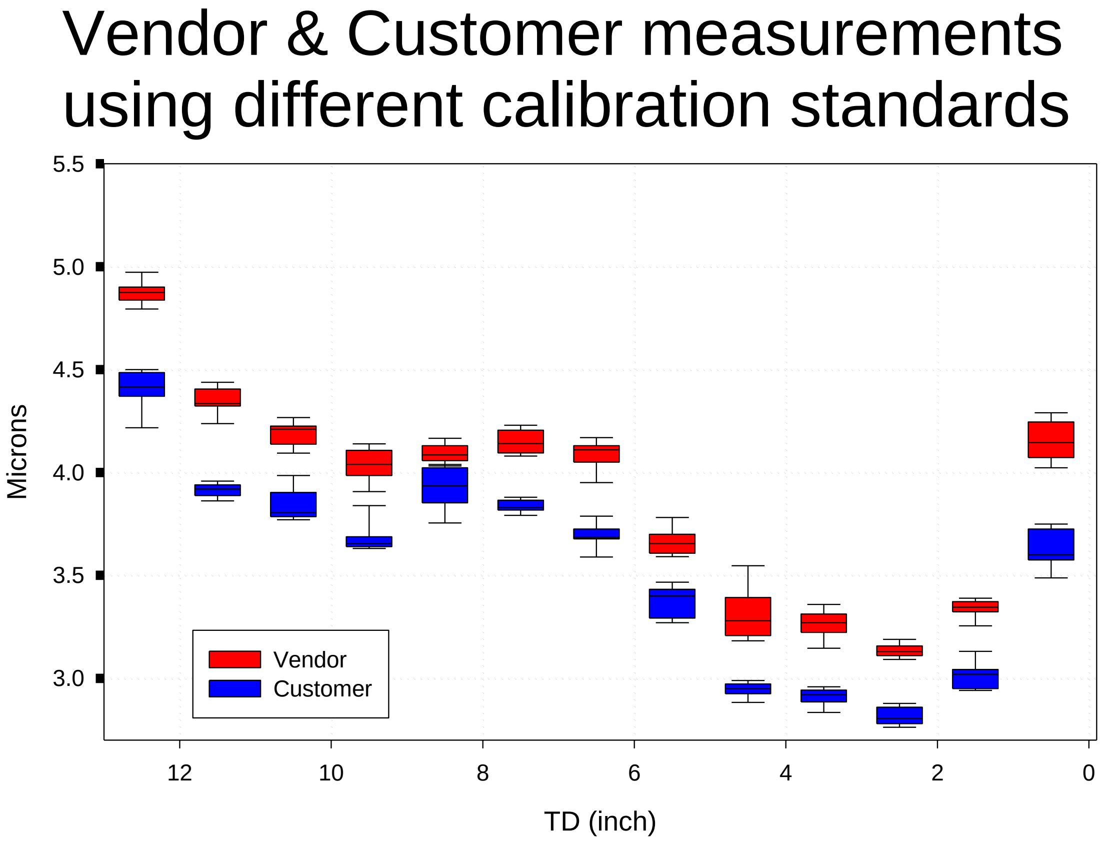


Figure 5

Since resistivity is the actual parameter being measured, and is inversely related to thickness, it is important to use calibration standards that bracket the area of interest.

The other factor to be aware of is the fact that electroplated deposits generally undergo recrystalization, or self-annealing, after deposition.[9] This relaxation typically takes on the order of a hundred hours at room temperature. It is advisable either to measure thickness only after such relaxation has taken place, or to set up a calibration curve using ‘unrelaxed’ standards. This behavior can be accelerated to 5-10 minutes at temperatures of 125-135°C. Errors approaching 20% in thickness measurement are possible if this detail is not managed.

An example of the effect of peeling freshly plated copper is shown in figure 6: a change in failure mode. The sample on the left has failed by the propagation of a ‘split film’ failure mode, in which the depth of the failure is sufficient to be visible to the naked eye on the foil side. The “annealed” sample on the right exhibits the normal cohesive failure. This mode is not apparent to unaided vision. (See figure 15 vide infra.)

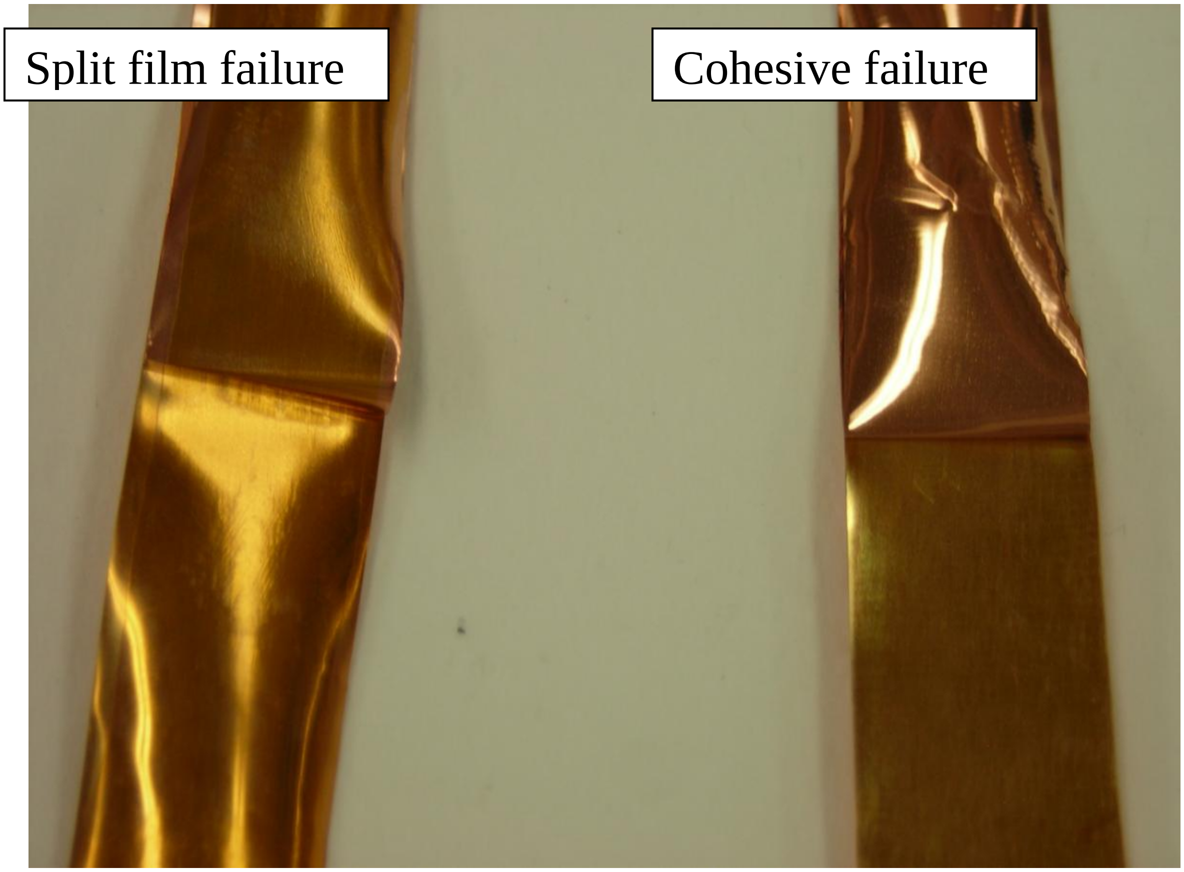


Figure 6

With these provisos on accurate thickness measurement of copper thickness, a vendor and a customer can talk meaningfully about specifications for peel strength of deposited adhesiveless FCCL.

So, you ask, if the customer and vendor agree on these thickness measurement details, the IPC method specifies peel strength values at 35µm copper in the requirements detailed in slash sheets from IPC4204. What is the big deal?

Firstly, the footnotes t1 and t2 are somewhat indistinct in that they appear to state that metal, base film and adhesive ***all*** must comply with the “greater than/less than” thickness provisions. Let us assume that the intent of the specification is to address the appropriate elements of a given FCCL configuration. In other words, let’s ignore the adhesive requirement for adhesiveless constructions. Then any construction with film <25µm and foil < 35µm should be judged by the requirements in column one.

Two questions arise. Is peel that dependent on film thickness? Figure 7 shows a control chart for method A (1/8” etched) peel strength for a deposited adhesiveless FCCL built on 12.5µm (half-mil) polyimide. The 1600 N/m (9.1 pound/inch) average peel strength for the five lots displayed agrees rather well with FCCL ‘B.’ That is, though these data were measured with 35µm copper, figure 4 would lead us to presume a relatively small contribution from the dielectric thickness, in the case of well-adhered deposited adhesiveless materials.

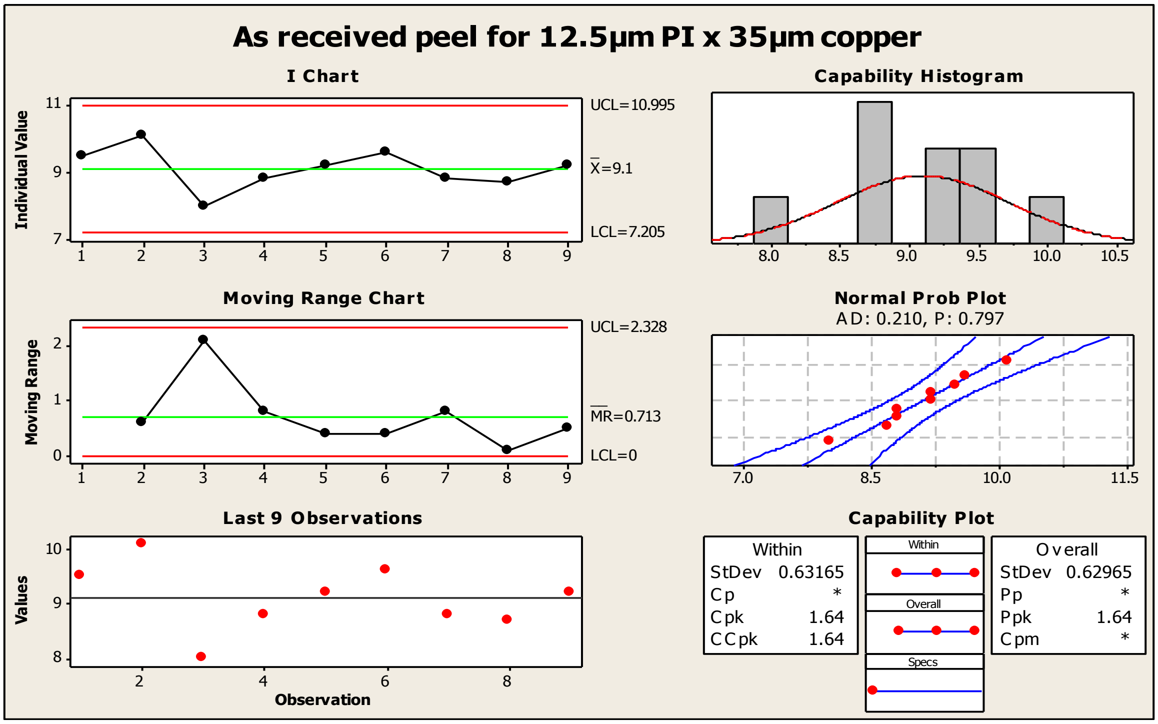


Figure 7

The second issue is that the footnotes only make a distinction of “less than/greater than” 35µm copper. It may comply with the slash sheet, but it would be misleading to plate to 25µm copper, and claim to meet the “less than 35µm” peel strengths. Why? Well, this is a holdover from the ‘foil mindset,’ and the quantized copper thicknesses available. Figure 4 clearly indicates that 20-25% higher peel strength would be expected for 25µm copper over “half-ounce” (17µm) plating. The intent is to specify peel strength with “half ounce” (17µm) copper.

Let us return to the issue of leaving the backside cladding in place to prevent tenting on the German wheel backside stiffener. Figure 8 illustrates that, for the example FCCL, copper thickness greater than 15-16µm results in the film tearing. The specification note, predating deposited adhesiveless clads, covered the subject well. In addition, the plot illustrates two other facts. First, below that 17µm copper thickness, we see the expected relationship of peel to copper thickness first illustrated in figure 2. Second, the data also indicates an offset between backed and unbacked samples even at these lower copper thicknesses.

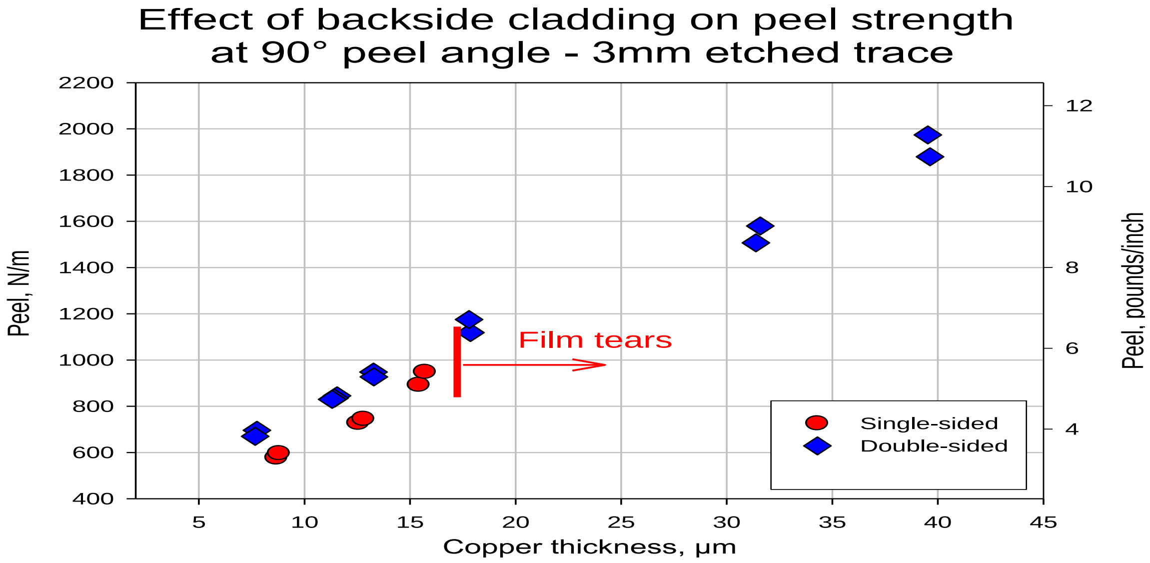


Figure 8

The same material was tested at 180°, yielding figure 9. The film does not tear at copper thicknesses >17µm. There is again an offset between the backed and unbacked conditions, even at 180°. It is possible that the adhesion of the PSA used to affix samples to the support influences these results.

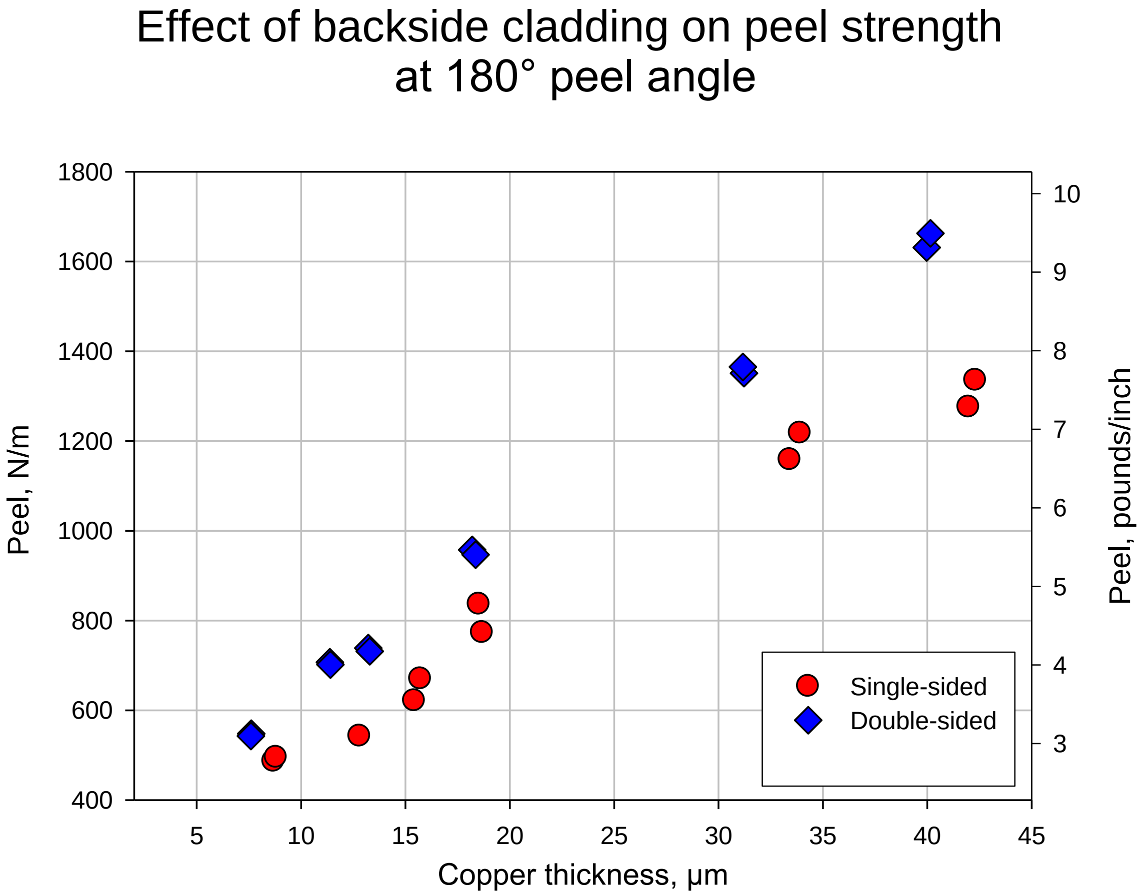


Figure 9

Surface finish is another example of a processing effect that can influence peel strength. The effect on peel strength of electrolytic gold plating on deposited adhesiveless FCCL has been described.[10]

Electroless nickel/immersion gold has become a popular surface finish, providing a solderable surface of uniform thickness that does not oxidize and offers some rigidity for good wire bondability. It is generally seen in the industry as a harsher chemical bath than the electrolytic variety.

It is commonly assumed that chemical attack is responsible for the reduction in peel strength that is sometimes accompanies ENIG plating. To be sure, the higher reduction potential of gold can lead to electrochemical reduction of hydrogen ions. Even radical anion formation of polyimide has been observed.[11]

Yet nickel has an oxidation potential similar to copper. The degradation of peel strength, when observed, begins in the nickel-plating step. What is going on?

A test was run in which a secondary imaging step resulted in ENIG-plating confined either to the very edges of an 3.2mm etched conductor, or is confined to the middle portion of the conductor. Figures 10 illustrate example traces.

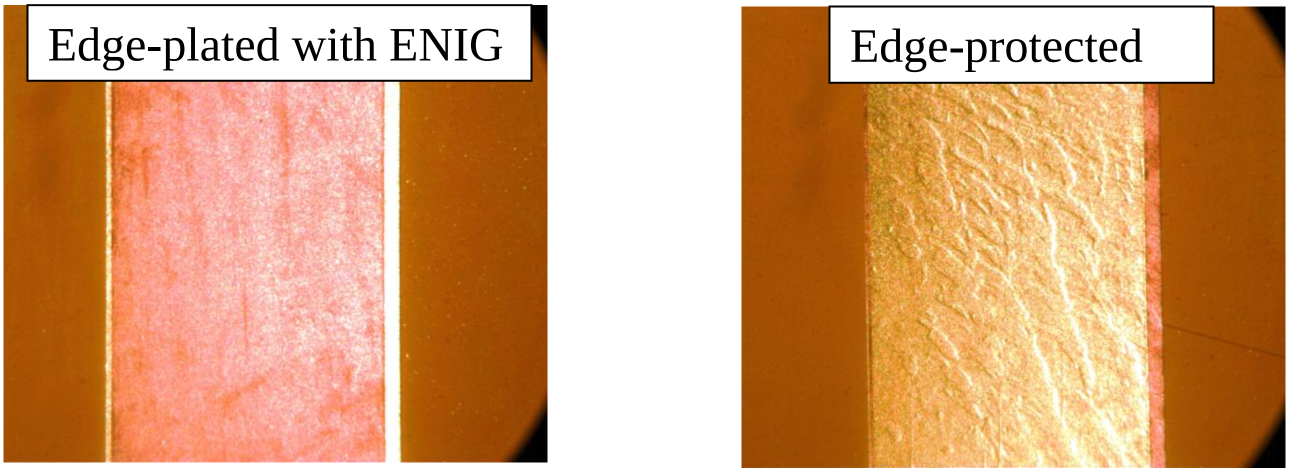


Figure 10

Since wicking is rarely observed to an extent beyond 2-3µm, it seems unlikely that chemical attack is responsible for degradation of peel strength with FCCL ‘A.’ If it is, the parts with edges protected should give significantly higher peel strength. However, if the influence derives from the effect on mechanical properties and subsequently, on the locus of crack propagation induced by the brittle nickel layer, the parts with exposed edges should exhibit higher peel strength.

The results of this peel strength comparison are displayed in figure 11.

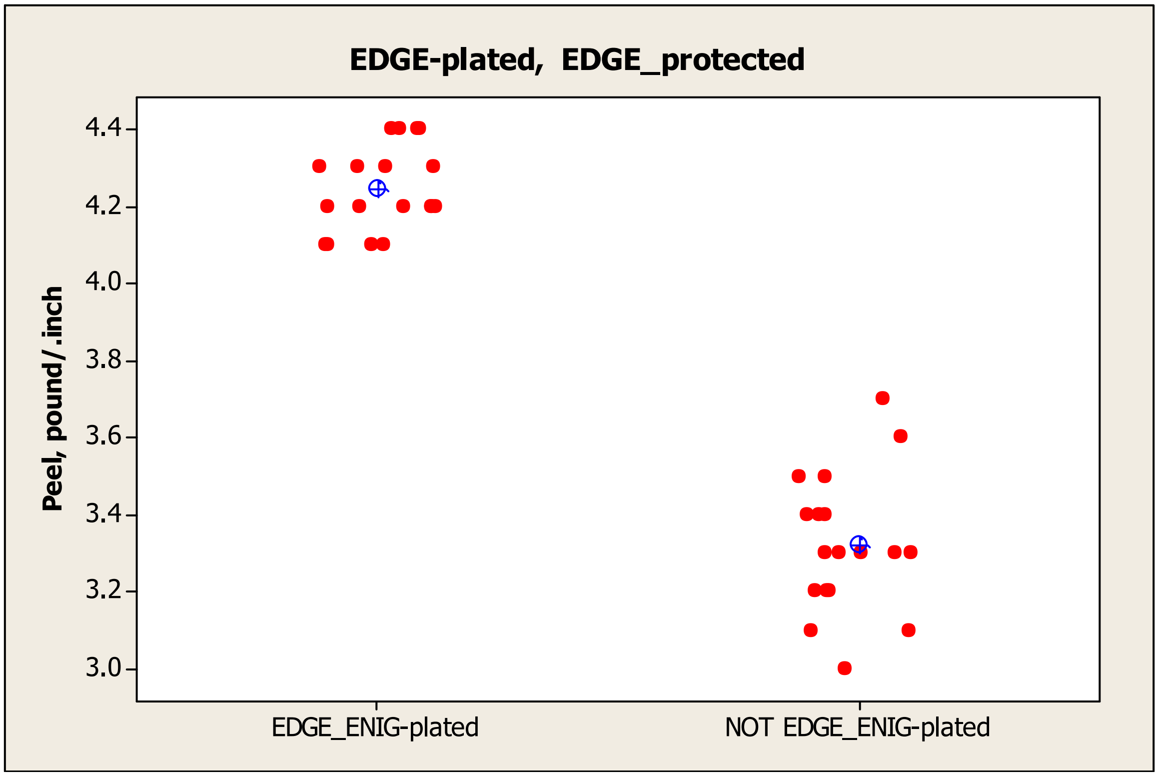


Figure 11

The foregoing test was performed on the ‘A’ type deposited adhesiveless substrate. Interestingly, when considering the higher adhesion ‘B’ version of deposited adhesiveless FCCL, we see no degradation in peel strength after ENIG plating. Comparing the data in figure 14 (below) against that in figure 3, we see an increase in peel strength after ENIG plating, due principally to the increase in conductor thickness.

The effect of surface-finish plating was tested in another way by comparing the peel strength of two structures of equivalent thickness, in this case 18µm copper, with a construction composed of 13µm copper plus 5µm of electrolytic nickel. Nickel has a modulus of 200GPa, ~140% higher than copper. Figure 12 compares the peel strength of these two constructions.

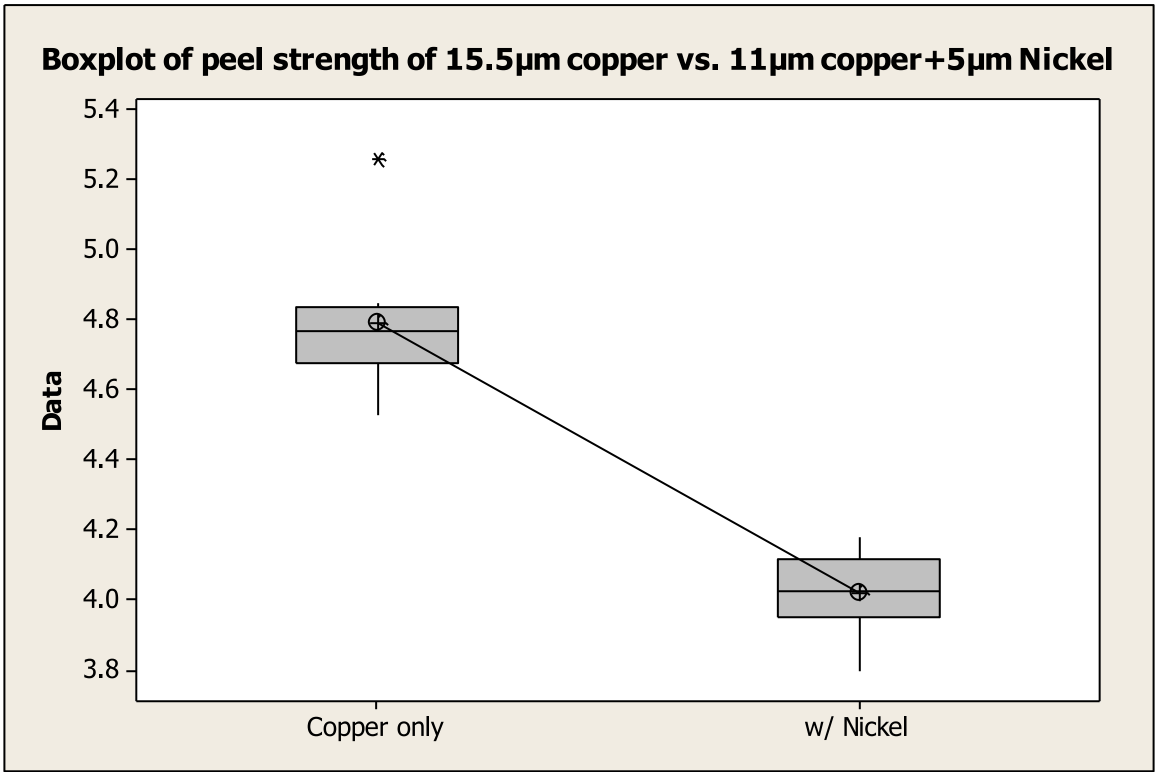


Figure 12

These results illustrate several interesting points. Surface finishes can reduce the peel strength measured. Yet, this appears not to be due to chemical attack (in the cases discussed here.) A change in the stiffness of imaged features must accompany the plating of nickel-based surface finishes. (As well, they like change the interfacial stress of conductors.) These data seems to suggest that the effect of this increased stiffness is to change the nature of crack propagation through the polyimide substrate, most likely driving it closer to the interface. This implies that the morphology of the polyimide film likely plays a role in this observed reduction in peel strength after ENIG plating.

Another difference in test configuration that deserves scrutiny is the configuration difference permitted by IPC, namely the etched conductor vs. the diecut specimen. Diecut samples will often exhibit tearing of the dielectric at lower stress levels than the corresponding etched specimens. In addition, more variability is observed with diecut specimens. Perhaps this is due to the boundary condition imposed by the nature of the specimen. An etched substrate can distribute the stress of debonding beyond the edges of the imaged conductor, whereas this obviously is not possible for the diecut sample. Figure 13 shows an example of the effect of this boundary condition.

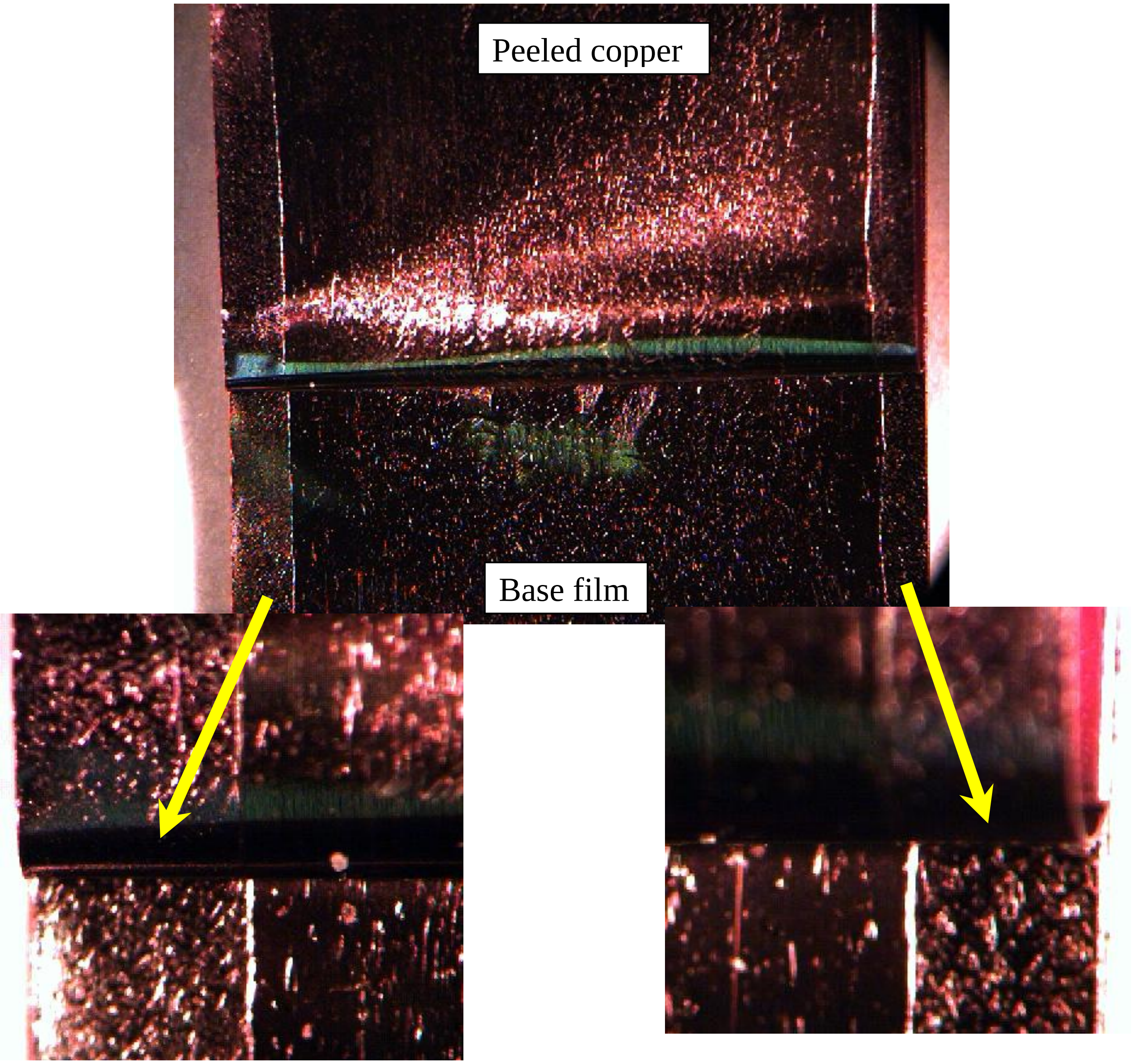


Figure 13

It would be good to be able to use the peel test to measure traces on fabricated parts, and know how to compare the numbers to what is reported with the standardized geometries of the IPC TM-650 2.4.9 etched conductors. Artworks were prepared with various compensations, and tests run on ~60µm wide lines over the range of copper thickness. Figure 14 displays the results. Figures 15-16 show the conductor and peeled substrate for the 8.0µm thick copper, and figures 17-18 show the test coupon built on 38.2µm copper.

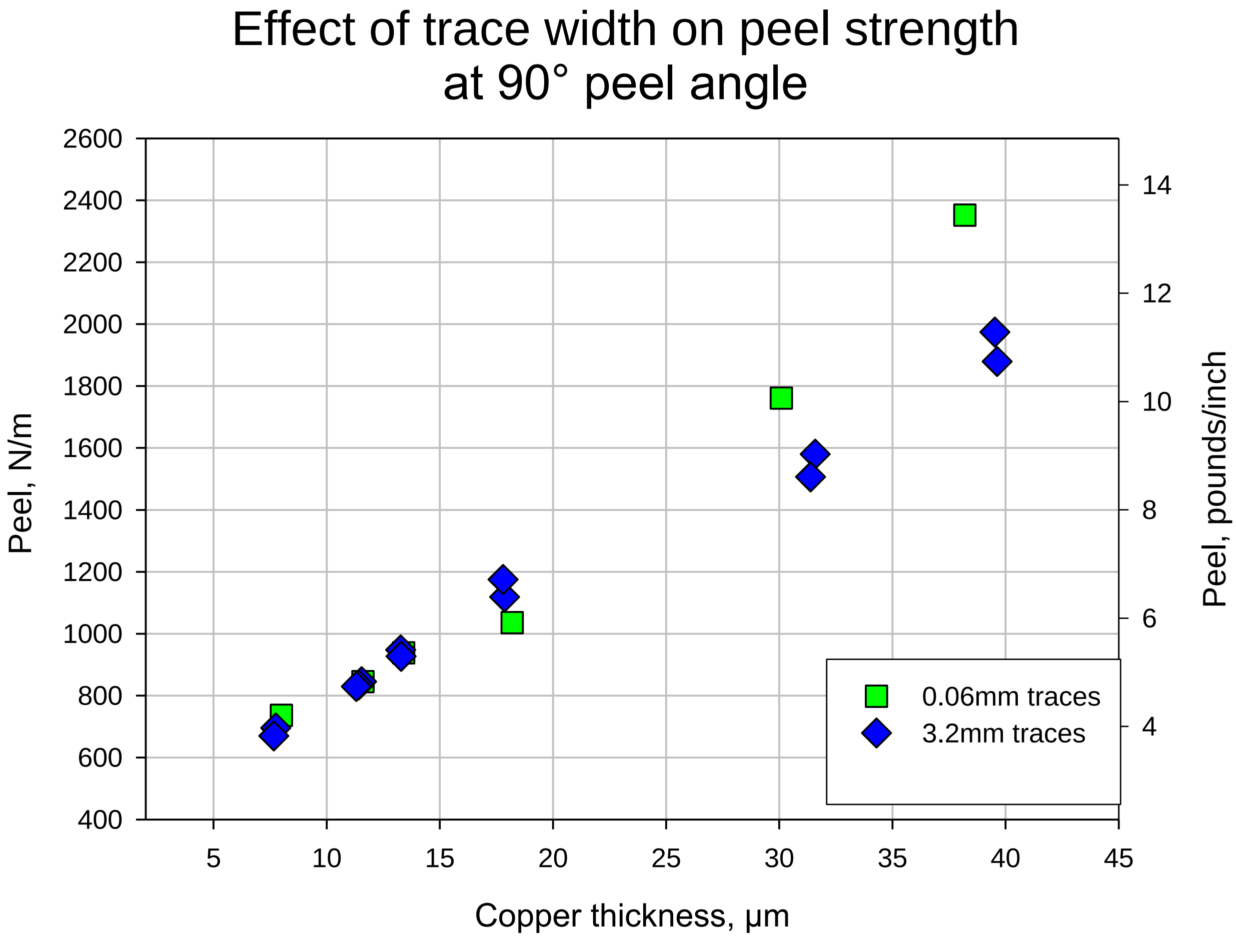


Figure 14

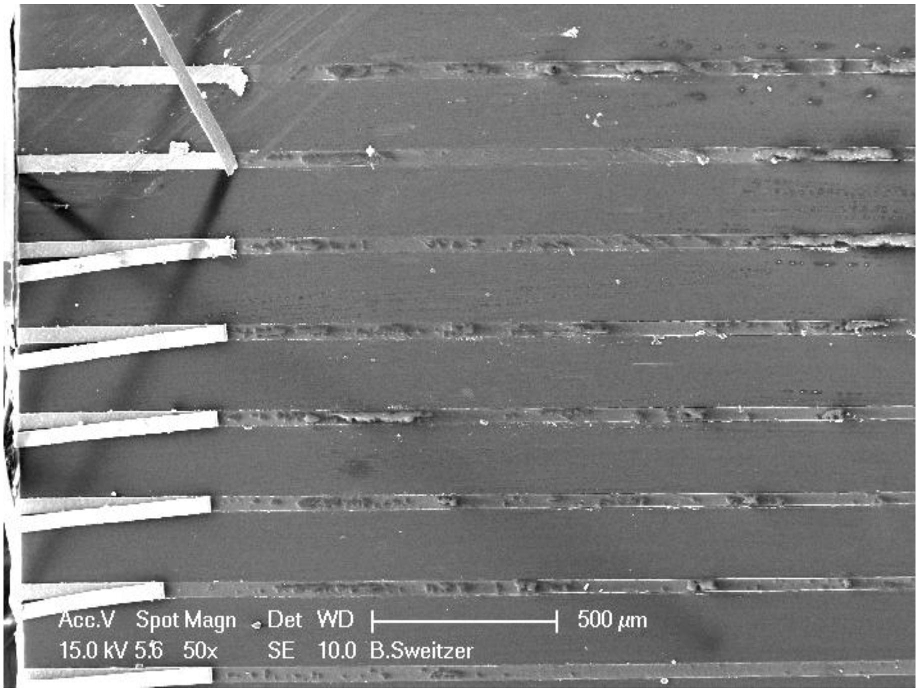


Figure 15

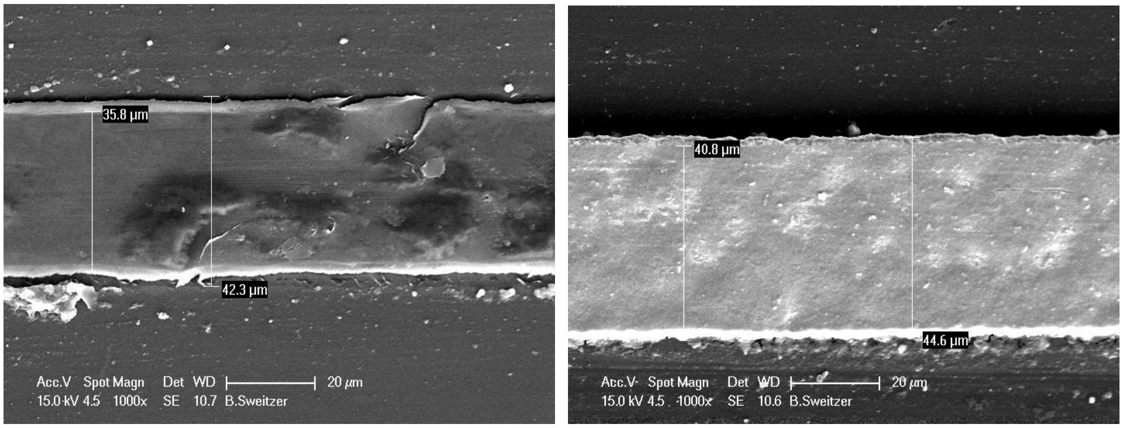


Figure 16

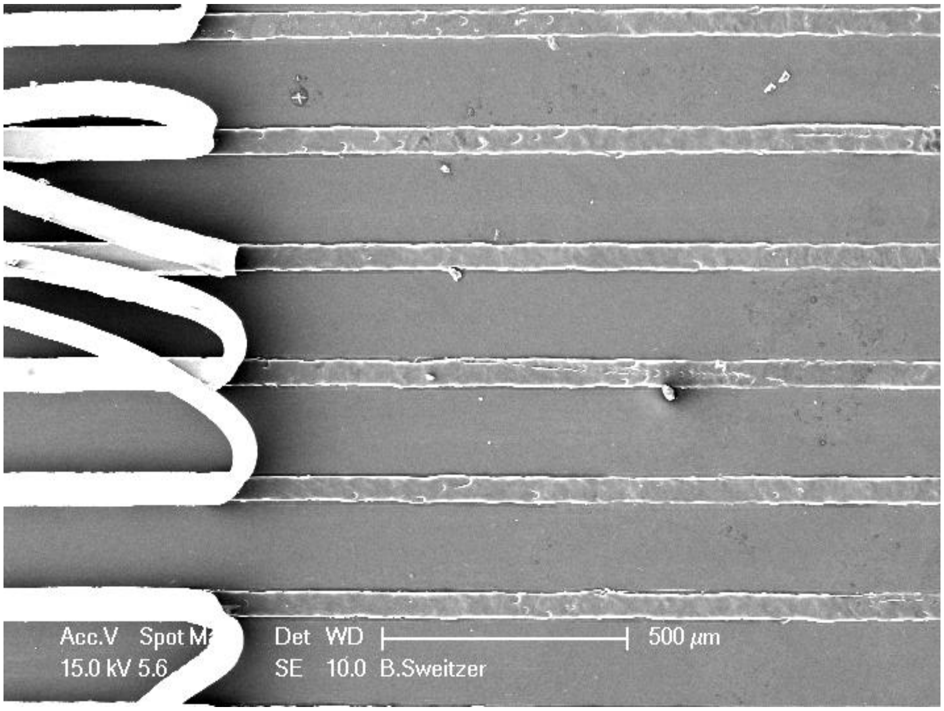


Figure 17

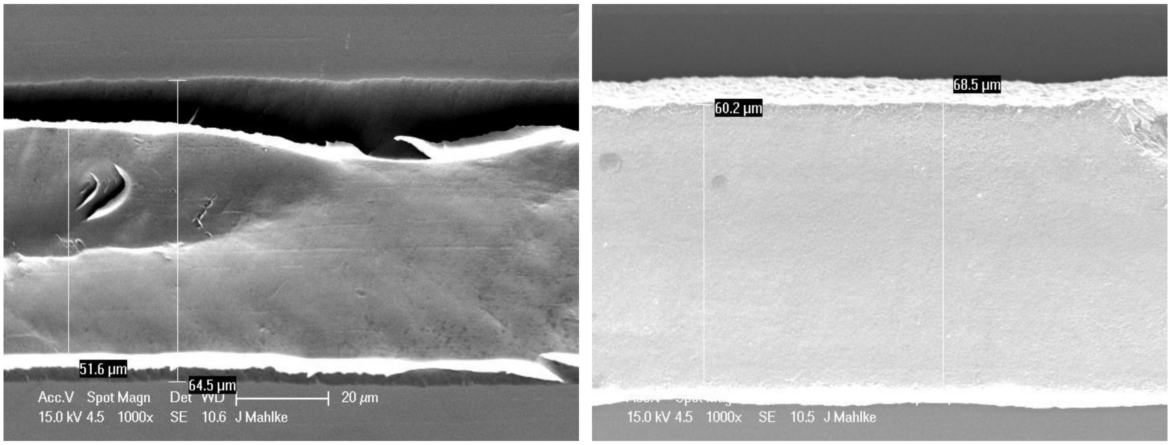


Figure 18

Which test matters?

When considering this question, three subjects deserve consideration: the intended application, the process flow, and the design. Automotive "under-the-hood" applications generally are tested for adhesion retention after one thousand hour exposure to 150°C, or to 85°C/85%RH conditions, or after one thousand thermal cycles from -50°C to 150°C. This would be overkill for a cell phone application, considering the product lifetime and the environment in which the device is expected to survive. On the other hand, much automotive circuitry has a HASL surface finish, while many cell phones receive either electroless or electrolytic nickel-gold. A validation test that the surface finishing step does excessively degrade adhesion is certainly worth considering.

If FPC is being fabricated with a polyimide ink overcoat, one should confirm that there are no deleterious effects from the extreme drying and curing steps, or at least, that these effects are understood and are acceptable.

At Sheldahl-Multek Flexible Circuits, we like to perform a battery of qualification tests, as follows:

As received
After solder float
After heat aging (168hr/150°C, or 72hr/210°C)
After 96hr pressure cooker exposure

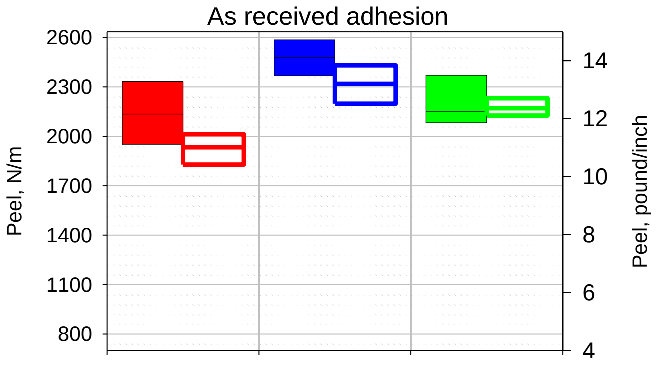
In addition, we will generally do these tests after ENIG plating as well.

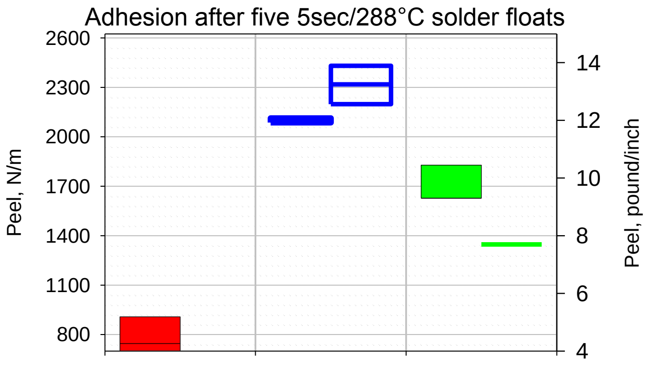
If a part is to be subjected to multiple lamination and/or soldering steps, it would be wise to examine the effect on adhesion. An example of the importance of this is found in a comparison we performed recently between cast-laminated-adhesiveless products and a deposited adhesiveless FCCL. This example also illustrates the importance of the IPC membership participating in review and upgrading of specifications.

The IPC peel-after-solder-float test method calls for the test to be performed after a five-second 288°C float. While this is somewhat more aggressive than that mandated by JIS, the ability to withstand multiple cycles at no lead reflow temperatures may not be fully demonstrated under these conditions.

In the comparison <u>five</u> <u>consecutive</u> solder float cycles at the 5sec/288°C condition were performed, with a two-minute cooling period after each. Figure 19 compiles the results of this comparison test, and illustrates something we were surprised to discover: one of the cast/laminated adhesiveless materials delaminated after five soldering cycles.

We noted with interest comparable adhesion strengths for the deposited material as compared to the non-deposited adhesiveless materials.





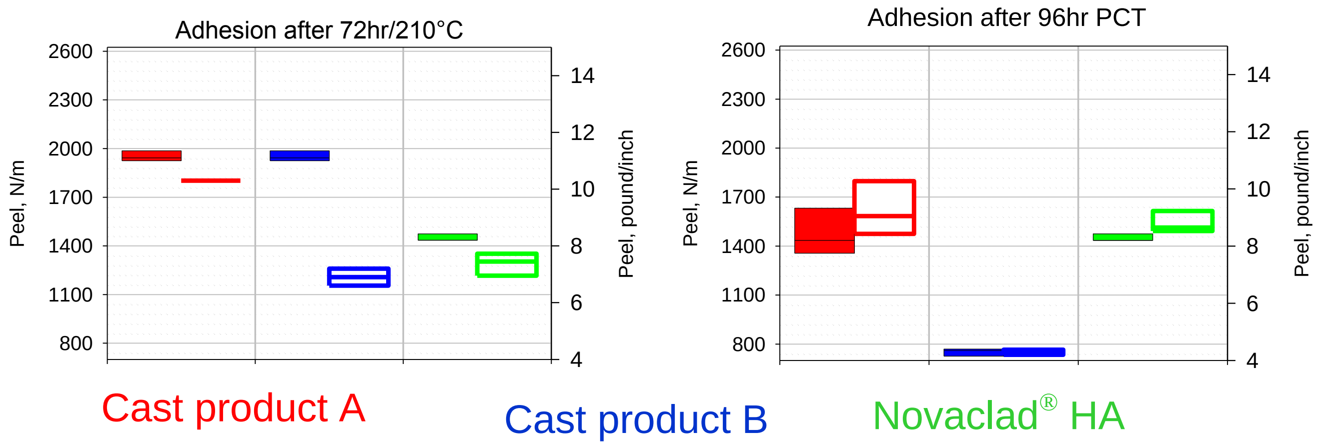


Figure 19

Finally, the circuit design has to be considered when judging what tests and acceptable values to require. As an example, a customer had been purchasing FPC built on adhesive-based FPC, but was dissatisfied with performance. After performing literally dozens of designed experiments on each of the component materials and the various process variables, we suggested trying adhesiveless construction, although we had never before produced the particular construction used in the customer's application. The performance improved by a considerable margin, and defects were reduced by an order of magnitude to an acceptable level. This was exciting news, but the customer perhaps got too excited: they promptly redesigned the part in such a way that the failure rate returned to levels originally seen. The only design that worked on the new improved (adhesiveless) substrate was the one first tested and validated (on the adhesiveless FCCL.)

Transferring designs from thin FR4 substrates to flex has led to trouble. For example, it was found that a design transferred in this manner produced inconsistent wire-bonding behavior on a 25µm polyimide base. After some careful study, it was found that wire bond pads with backside traces running partially underneath the pad bonded poorly, while those pads with a solid copper structure or with no copper behind the pad wired in a consistent manner. In contrast thin FR4 apparently was rigid enough not sensitive to this design difference.

Failure modes

Deposited adhesiveless clads are characterized by cohesive failure within the film. Occasionally, a split film mode is observed, as shown in figure 6.

Figure 20 below illustrates the failed film surface for lower adhesion product.

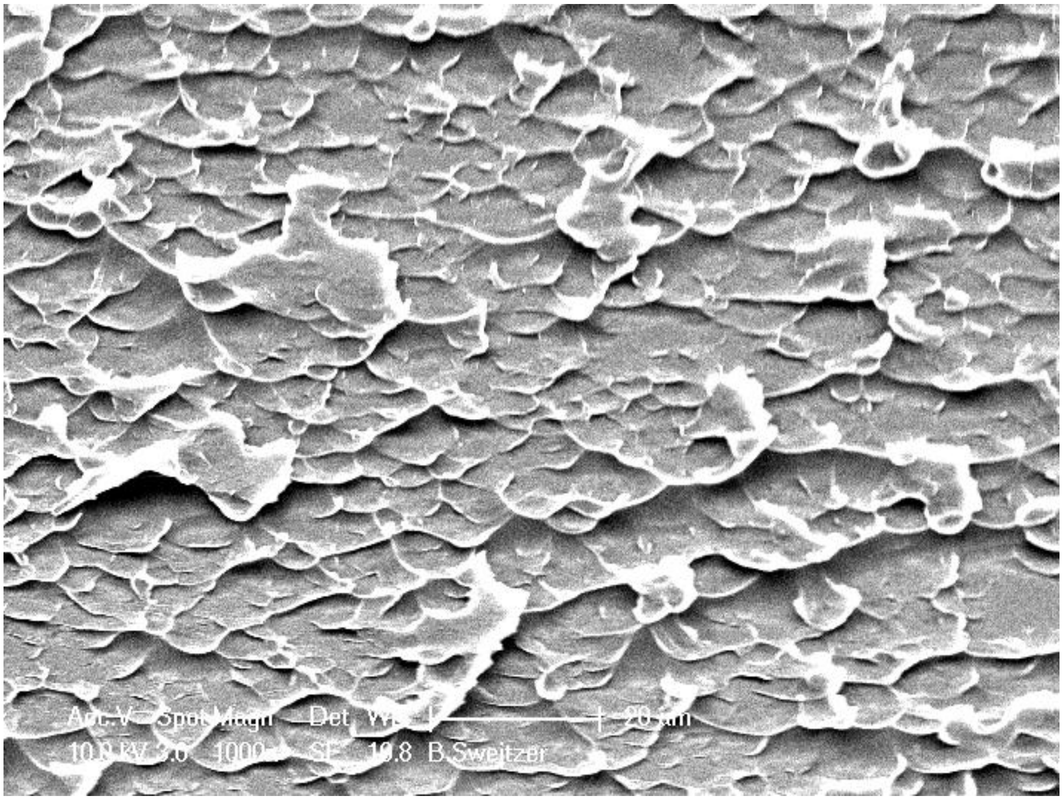


Figure 20

It may seem surprising that a cohesive failure with such evident disruption would accompany a material with lesser adhesion. Figures 21-22, of the higher adhesion ‘B’ product, seems to evidence a toughening of the polyimide near the interface.

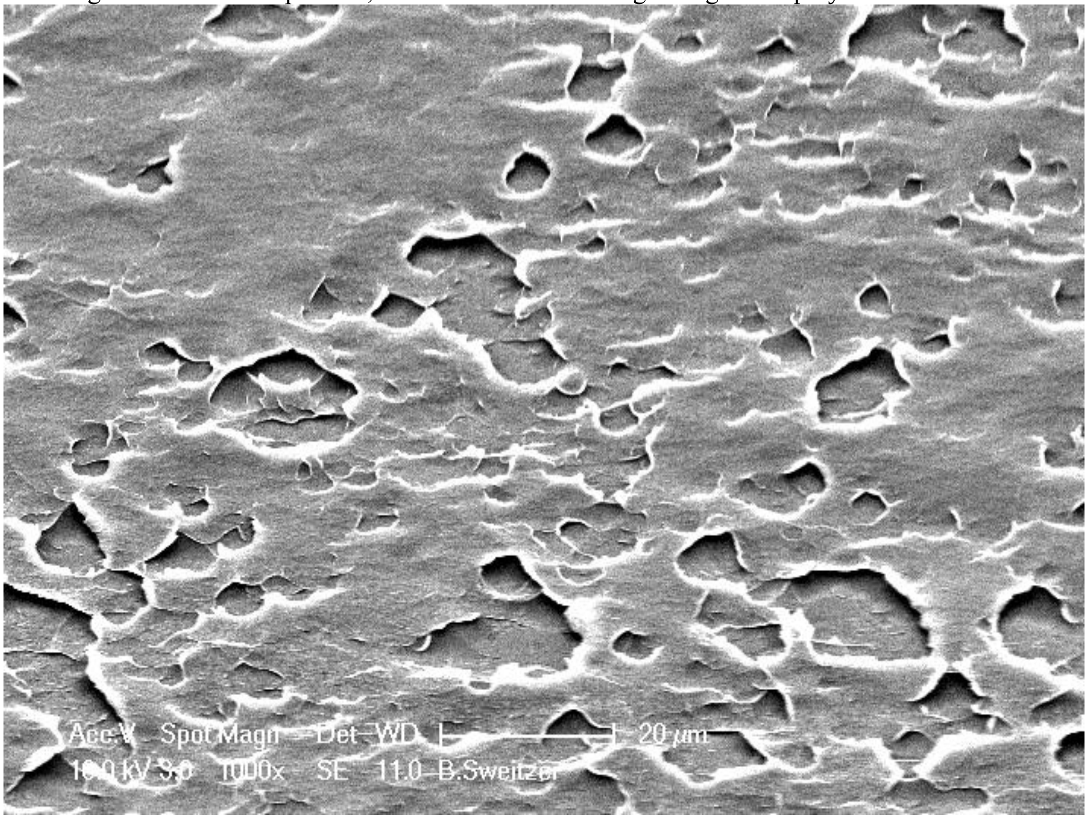


Figure 21

At higher magnification, the disruption and cohesive failure within the PI can be seen in figure 20. In addition are observed what appear to be small crystallites within the polyimide structure.

Figure 22

Other types of adhesion tests

We mentioned that peel testing is performed primarily at 90° angle, and to a lesser extent, at 180°. Certain FPC fabricators have advocated a T-peel geometry as shown in figure 23. The stated reason for this preference is a perception of less variability in results. While the IPC has reviewed the T-peel, the approach has not gained acceptance in the industry.

Figure 23

Another way of measuring adhesion is an out-of-plane tensile test. A specimen of FCCL is glued to a ceramic support. A metal stud is then glued to the copper surface and the stress is applied on an axis normal to the sheet of FCCL. This method is not particularly useful for flexible materials since, by their very nature, flexible materials experience significant stresses that are not perfectly normal to the plane of the material. Cases have been encountered where 10,000 psi tensile strength is measured on an FCCL that can be delaminated with a flick of a fingernail. Figure 24 is a schematic of the tensile test.

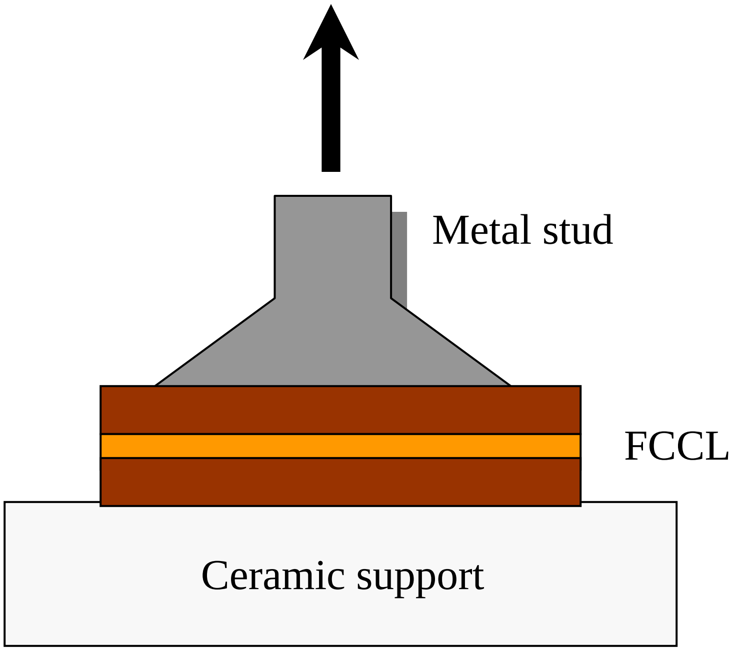


Figure 24

Figure 24 shows the witness mark of the shear at higher magnification, which is the "trench" formed in the polyimide.

Another method used in some cases is a shear test. The force required to initiate this failure is reported. The failure happens to propagate as a peel, since the movement of the anvil begins a distortion of the metal that causes the metal to roll up like a sardine can lid. Figures 25-26 relate to the shear test. The first shows a pad to which a shear stress has been applied from the lower right-hand corner, resulting in a rupture that resembles a trench.

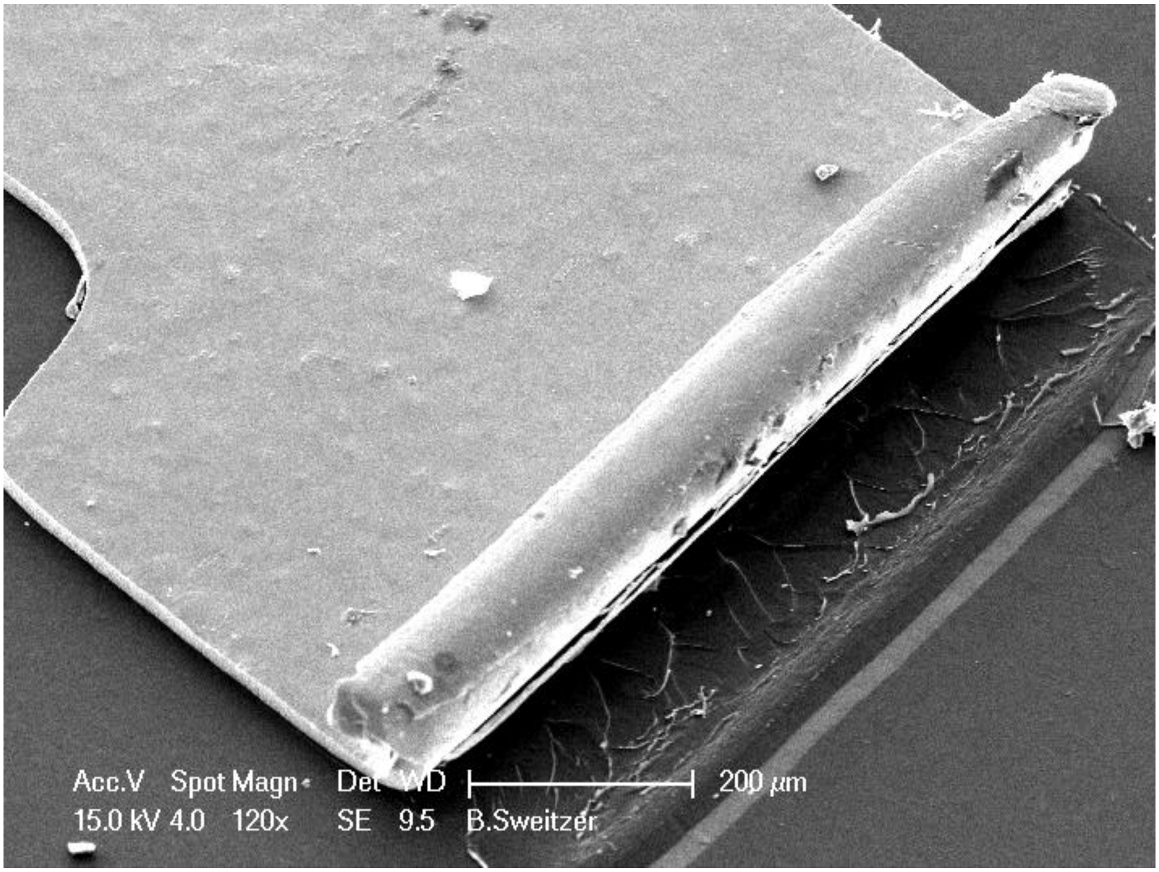


Figure 25

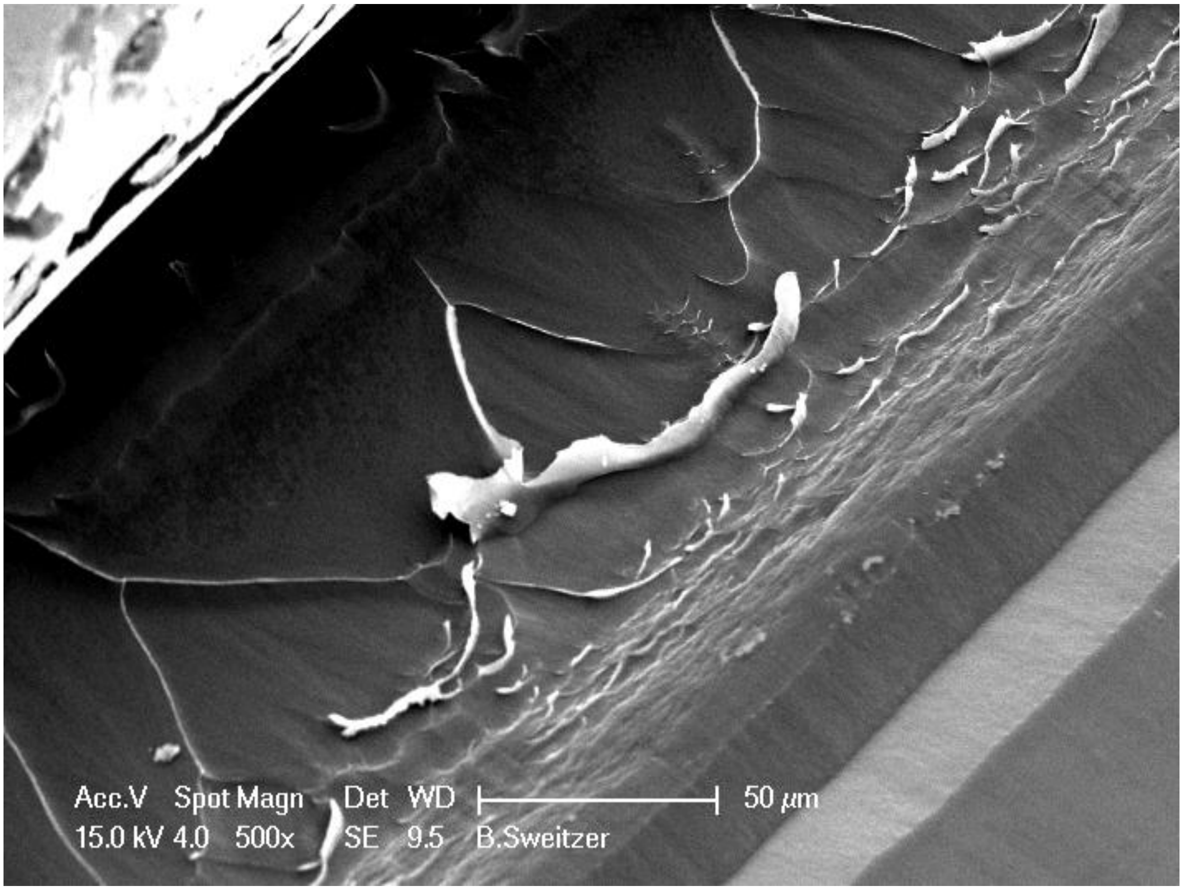


Figure 26

Other tests that have been proposed include microindentation testing, in which a stylus is brought into contact with a coated substrate and the force to cause to crack and finally to debond the coating is measured. This method is often used with thin film coatings. It has value for measuring adhesion of coatings on rigid substrates, such as silicon, but yields variable results for coatings on lower modulus supports such as polyimide.

For brittle coatings such as ITO (indium tin oxide,) application of inplane tensile stress will lead to cracks in the coating. These cracks develop and propagate with a separation distance that is indicative of the adhesion of the coating. This “stretch & crack” technique is often used for brittle coatings, such as ITO on plastic films or magnetic films.[13,14] The high ductility of copper makes this method unsuitable for FCCL, although it has been evaluated on sputtered copper on polyimide.[15]

Another method of adhesion measurement for high tensile/low elongation coatings has been described where a hole is formed only in the substrate of the coated material. By application of compressed gas to this void, a blister develops in the coating. The relationship between the force applied and the size and rate of blister development allows one to derive a measurement of adhesion.[16]

Conclusion

The peel test, while bemoaned for its shortcomings, remains a standard for judging adhesion of FCCLs. The movement to deposited adhesiveless FCCLs, with the much wider range in metal thicknesses, introduces a note of complexity into the peel test not formerly present with foil-based products. When comparing peel results, the range of metal thickness needs to be considered, as well as how that thickness is measured. Specimen configuration is one of a number of other items that deserve consideration when comparing and contrasting peel data.

People have thought that deposited adhesiveless FCCLs exhibit inferior peel strength when compared to their cast or laminated ‘cousins.’ Data presented herein shows this not necessarily to be the case. Being able to interpret a peel test on fabricated interconnects permits one to better answer the question, “how much peel is enough?” Cognizance of the factors influencing peel adhesion is crucial to making an informed decision.

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