

A New Method for Measuring Conformal Coating Adhesion

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Abstract

Coating adhesion has been a difficult property to measure, and the industry has made do with a scratch test that is only capable of qualitative tests. NPL with industrial partners, have developed a tape peel test that can be applied to the PCB or component surface. The choice of tape is critical in achieving complete wetting of the fabric, and good adhesion to the coating. The tape is applied with liquid coating to the substrate, and then cured, leaving a flying unbonded section for clamping on during the pull test. The method shows clear differences in adhesion between different coating types: acrylics, polyurethanes and silicones. The effect on coating adhesion of surface cleanliness and the cure state of the resist were investigated. Coatings were generally observed to perform well with these problems. However, coating adhesion to components and some resists proved much more variable, with some coatings failing to adhere to problematic components. Surface energy measurements using a wetting angle technique were also used and compared with the peel data.

Introduction

Coatings are increasingly applied across a wide range of applications to offer environmental protection [1,2,3], and some of these are demanding in terms of the protection offered by the coating in preventing permeation of contaminants [4,5,6]. Of particular importance is that the coating remains adhered to the substrate. Loss of coating adhesion is primarily a function of contaminants within the assembly process, and hence a method that can quantify the adhesion within the assembly environment is needed. Currently there are two methods available. One is the scratch and peel test as described in [7,8,9], but the method is operator dependent and not quantitative. The second approach is the use of dyne pens to assess substrate surface energy, and is based on a visual inspection of the pen marks. This again is not quantitative, dependent on operator interpretation, good condition of the pens, and no compatibility issues between the pen inks and the contaminant surface.

Here we introduce a peel test, based on a tape being cured in the coating and then peeled from the substrate in an instrument, with additional details given in [10]. The introduction of the tape should not affect the cure process and have a strong cohesive strength within the coating, so that during the peel the tape lifts the coating from the substrate, rather than pull the tape from the coating.

Approach

In Figure 1 a cross section schematic is shown of a fully immersed fabric tape in a coating, and an image of a prepared sample on a laminate substrate. Excess coating is squeegeed off the fabric tape to result in a total thickness of $\sim 200\mu\text{m}$.

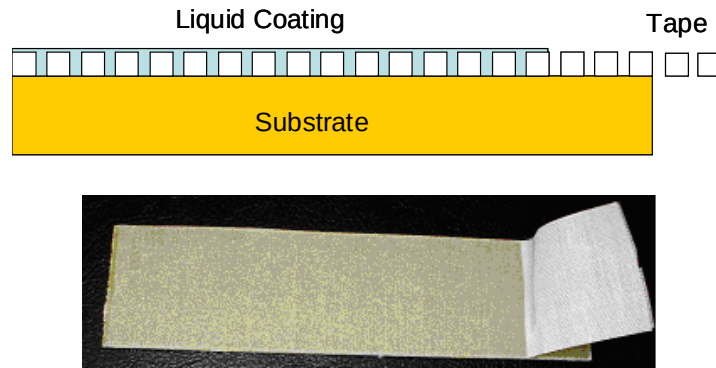


Figure 1: Schematic and picture of test sample

Once cured the sample was peel tested, and we achieved this with a Dage bond tester, as shown in Figure 2, although any suitable peel tester could be used.

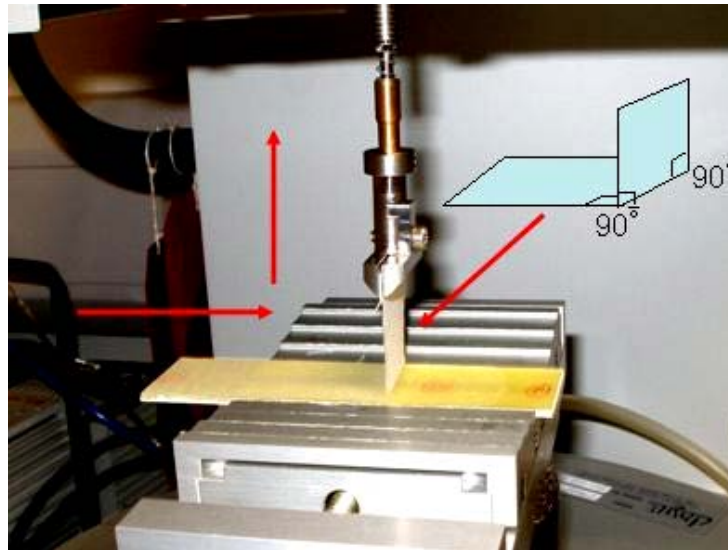


Figure 2: Tape peel test

Typical results from this test are shown in Figure 3.

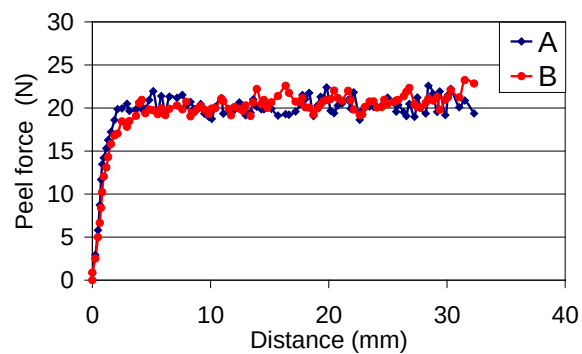


Figure 3: An example of peel tests

A significant issue with this approach was the tape material, and a number of meshes: stainless steel, brass, nylon, polyester, and polypropylene; and a number of cloths were evaluated: glass, polyester, cotton, silk, 20% polyester + 80% cotton, 65% polyester + 35% cotton, 70% polyester + 30% cotton.

The peel force results from tests on these tapes (25mm width) with an acrylic coating are presented in Figure 4 and 5. The data reveals that fabrics generally out performed the meshes, and this can be attributed to their fibrous construction, providing high surface area, and rough fibres, for adhesion to take place on. Also of importance is the open area of the tape, as all coatings require good access to the atmosphere for curing. Hence, the 65% polyester and 35% cotton mix fabric with 15% open area, which gave the highest peel force, was selected for all the further tests.

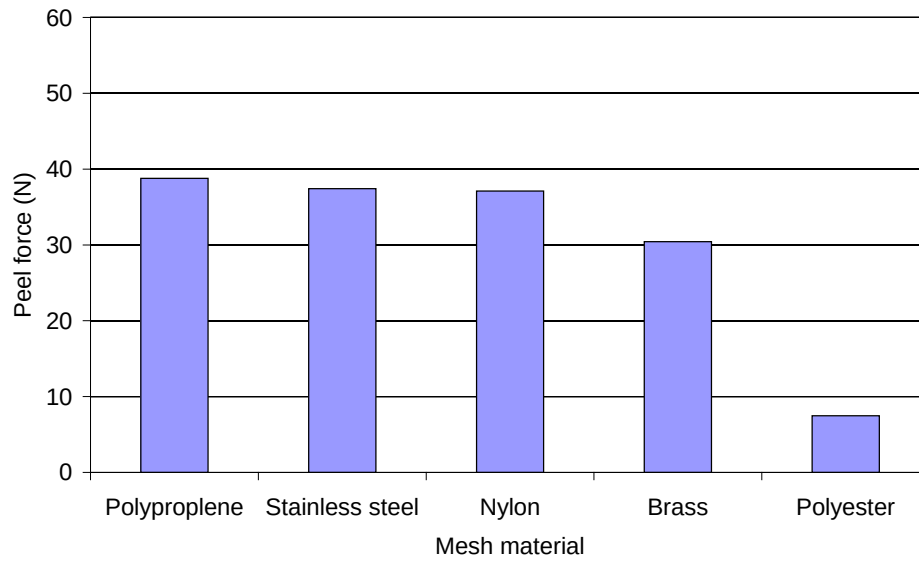


Figure 4: Average peel forces for different mesh materials

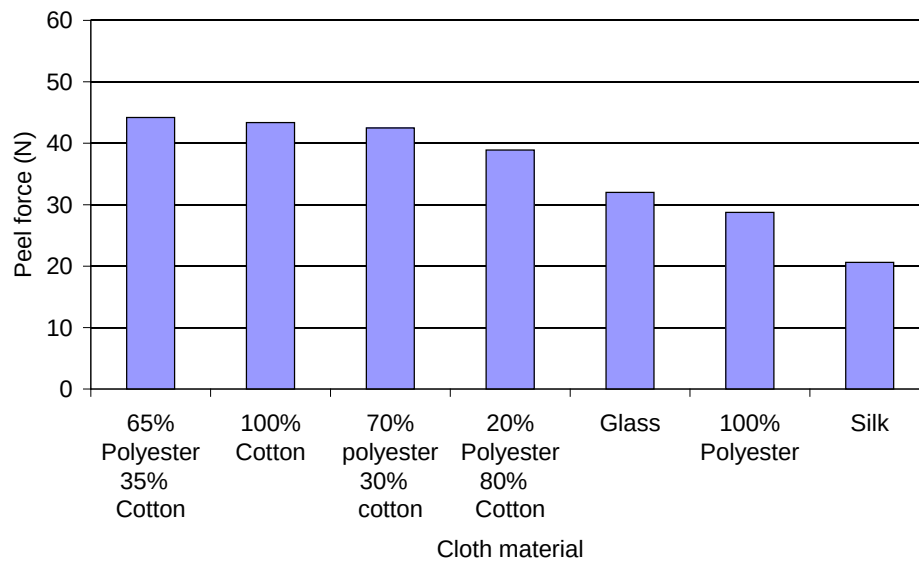


Figure 5: Average peel forces for different fabric materials

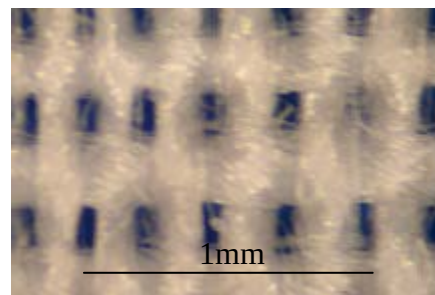


Figure 6: Tape material (65% polyester plus 35% cotton fabric)

Effect of peel speed

The peel speed could be varied from 0.5 to 1.5mm/s with this Dage instrument, and the effect of this on the three coatings is shown in Figure 7, again using a laminate substrate.

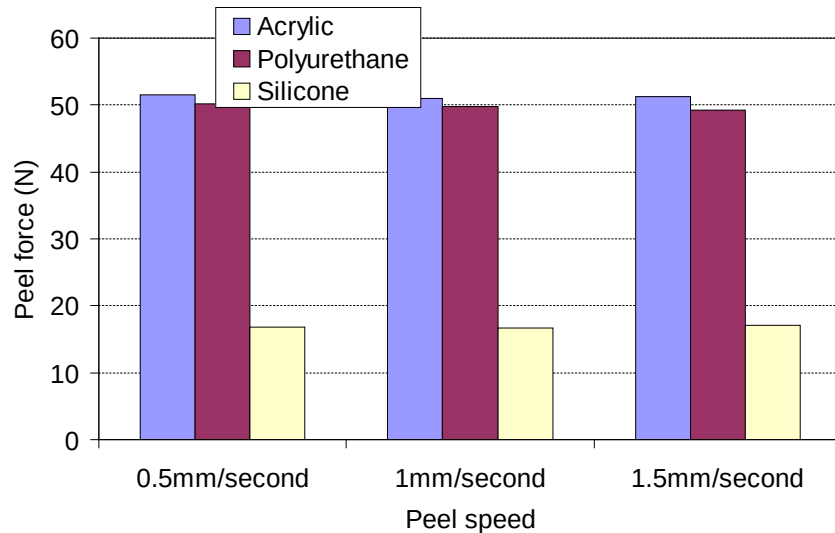


Figure 7: Average peel force for three type coatings at different peel speeds

These results show there is little dependence on peel speed over the tested range, but again the low adhesion strength of silicones is apparent. For the following work, a 1mm/s speed was used.

Tape peel width

The peel forces with different tape widths (5, 10 and 25mm) for three coatings are plotted in Figure 8 respectively. The dotted lines represent the backward extrapolations of the 25mm tape width values, to check if the force decreases pro-rata with width. The results show that the force broadly follows tape width, but specifically at 5mm the force is noticeably higher than expected, and this can be attributed to edge effects. Therefore, it is recommended that a minimum of 10mm tape be used, if possible.

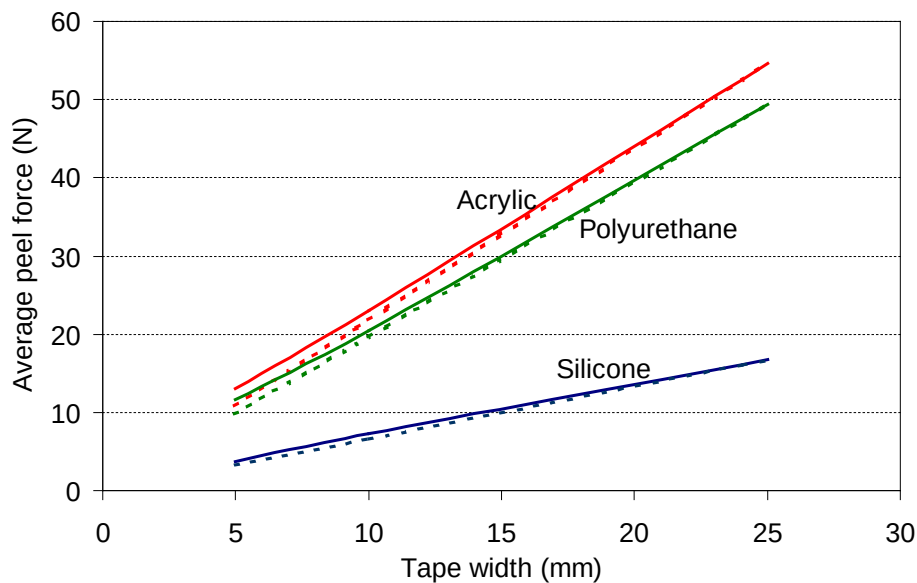


Figure 8: Measured and extrapolated peel force from different tape widths for three coatings

Cure conditions

The sensitivity of the peel test to the cure state of three coating materials was investigated, and the test conditions are tabulated in Table 1.

Table 1: Cure temperature and time for three coatings

Acrylic A		Polyurethane		Silicone	
Ambient Hour	72°C Minute	Ambient Hour	80°C Minute	Ambient Hour	Ambient (hour) + 80°C (Minute)
1	20	16	30	24	24 hour + 20 minute
2	40	40	60	48	48 hour + 20 minute
4	70	66	90	72	72 hour + 20 minute
8	120	96	120		
24		144			
48		216			
96					

Using the same test procedure as before, i.e. 25mm tape width, 1mm/s peel speed and a peel distance of 35mm, the following results were obtained, and are presented in Figure 12. All peel test values are for an average between 5 and 35mm of peel for each of two tests.

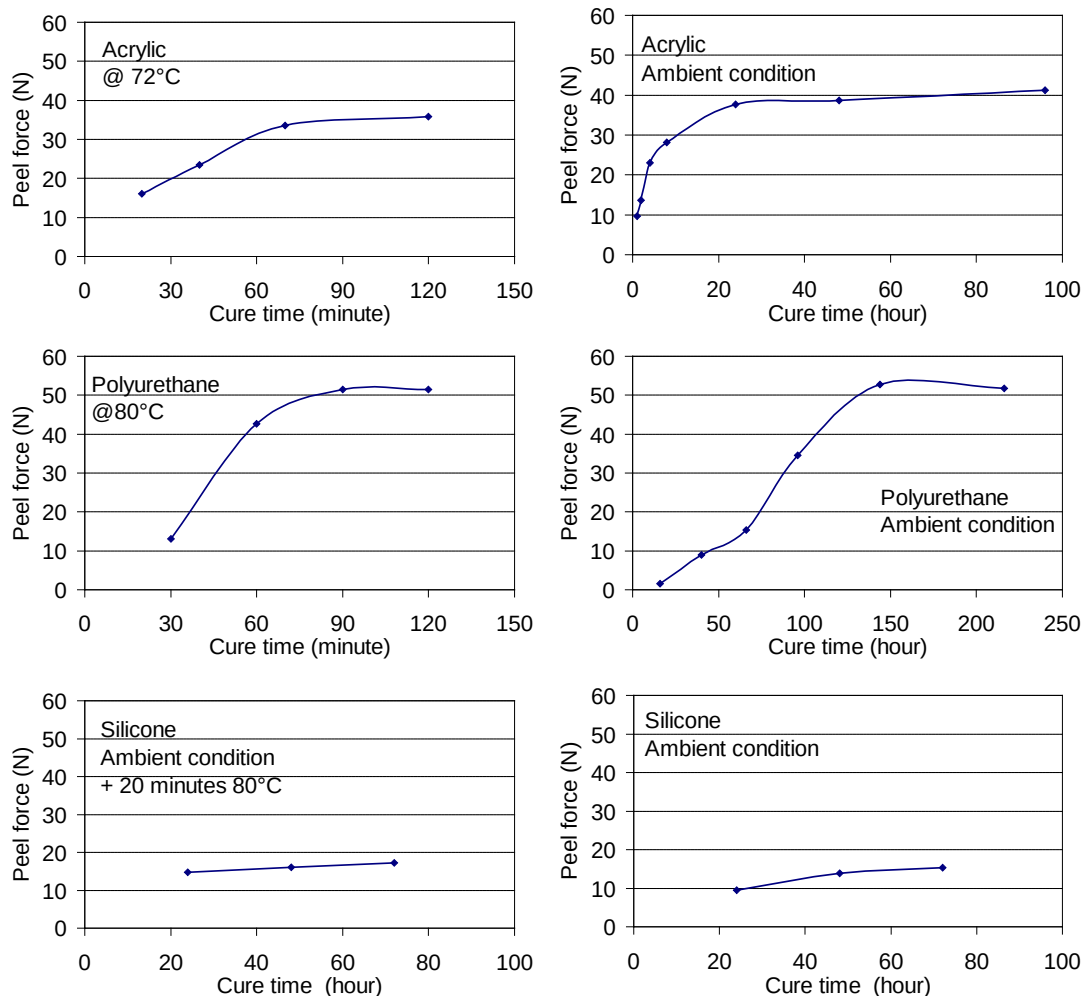


Figure 9: Average peel force from different cure conditions for three type coatings

The results in Figure 9 clearly show the role of cure in improving the adhesion, except for the silicone, where the failure was at the tape interface. In some instances, the cure at elevated temperatures produced bubbling under the tape. This is attributed to solvent loss issues, where the tape restricts this process. However, elevated temperature definitely accelerates adhesion, and the test method provides a useful means of checking any given cure schedule. From this work, it was decided to apply the following cure schedule for the three coating types for subsequent work in this study, summarised in Table 2. The mixed cure schedule was to allow the initial solvent loss to occur and prevent bubbling during the 80°C cure.

Table 2: Cure condition for three coatings

Acrylic	Polyurethane	Silicone
24 hours in ambient + 30 minutes @ 80°C	24 hours in ambient + 60 minutes @ 80°C	24 hours in ambient + 20 minutes @ 80°C

Contamination effects on acrylic coating adhesion

With a clean PCB, the failure always occurred at the tape/coating interface, instead of the coating/substrate interface, reflecting the strong adhesion of the coating to the substrate. To establish the test response as the surface energy decreased, attempts were made to contaminate the surface. Hence a number of common commercial products were trialled, and these included solvent and water based solder fluxes, various petroleum based greases with different melting points, a grease with added slip agents, silicone lubricants and a silicone release spray. All of these failed to impact significantly on the adhesion force, which can be possibly attributed to the washing action of the co-solvents in the coatings. However, common surfactants used for general cleaning were found to significantly impact adhesion. For these trials, the laminate substrate was contaminated with a common proprietary surfactant, which was dissolved in deionised (DI) water at three different concentrations, as listed in Table 3. For each test, 1.0ml of the contaminant solution was dispensed onto a 25 x 100 mm laminate sample. The sample was then dried at 100°C oven for 5 minutes before applying the coating with the tape. Two samples were tested for each contamination level.

Table 3: Contamination levels of surfactant

Coating	Contamination (% in DI water)
Acrylic A	None
	0.5%
	1.0%
	3.0%

The average force for each different contamination, are presented in Figure 10. The substrate and peel tape were inspected under a microscope after testing, and the results are shown in Figure 11.

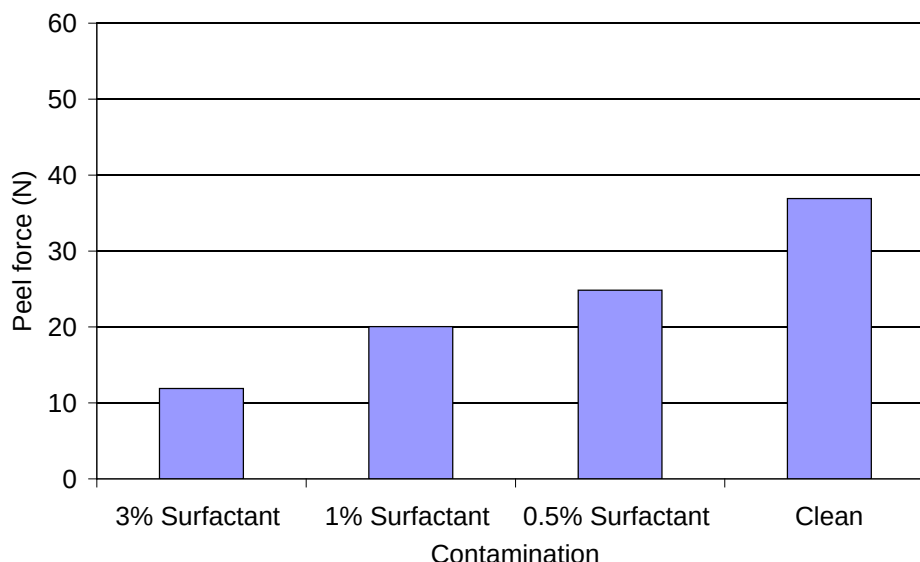


Figure 10: Average peel forces for different contamination samples

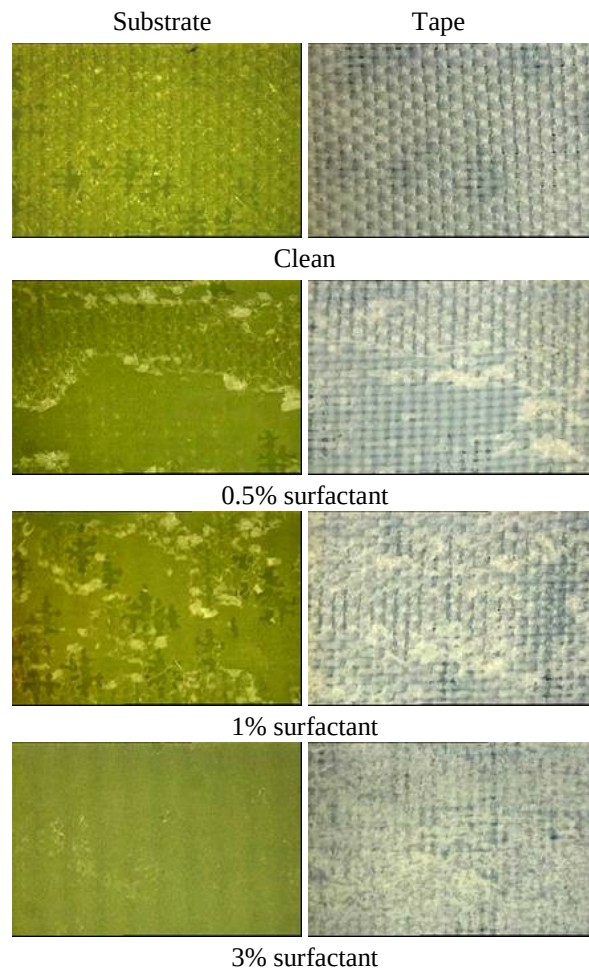


Figure 11: Substrate and peel tape for different contamination samples after testing

The results in Figure 10 and 11 clearly demonstrate that peel force decreased with increasing contamination, at that the failure mode changed from the tape/coating interface to coating/substrate interface. As the contamination on the substrate increased to 3% surfactant, the peel force dropped from 37N to 12N for the clean board. At the intermediate contamination levels, a mixed failure mode was observed, with failure occurring more at the coating/substrate interface with increasing contamination. This suggests that the test method can discriminate between different surfactant contamination levels.

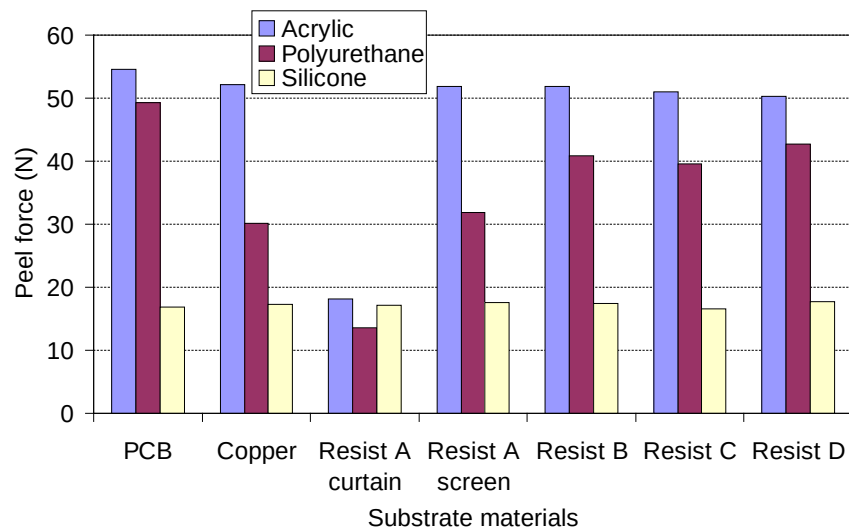


Figure 12: Average peel force from different materials for three coatings

Effect of resist

Several resists were evaluated, Coates XV501T-4, Coates XV501 CAWN1287, Huntsman Probimer 77 and Electra EMP100. All resists were applied by screen printing except one which was also curtain coated. The four resists were arbitrary named A to D. These resists were supplied to NPL from PCB fabricators. The same test method was applied as before and the results are shown in Figure 12 along with the coatings on bare laminate and copper clad laminate.

The failure mode is given in Table 4.

**Table 4: Failure mode from different substrate materials for three coatings
(T: tape/coating interface, S: coating/substrate interface)**

Substrate Coating	PCB	Cu	Resist				
			A Curtain	A Screen	B	C	D
Acrylic	T	T	S	T	T	T	T
Polyurethane	T	S	S	S	T+S	T+S	T+S
Silicone	T	T	T	T	T	T	T

Again the results were not informative for the silicone, with low forces and the failure at the tape/coating interface. The acrylic out performed the polyurethane with high adhesion forces across the samples with the failures occurring predominately at the tape/ coating interface. There were many failures at the coating/substrate interface with the polyurethane, which showed generally poorer adhesion to resists than the acrylic. In addition, one resist (Resist A curtain coated) gave poor adhesion values revealing a more systematic problem with this coating. Hence, the technique shows the possibility of quantifying the adhesion between resists for some coatings.

Effect of components

A random selection of plastic encapsulated IC components, see Table 5, were purchased and the adhesion force measured using the same method as before, except a 10mm tape was used, and therefore the forces are typically 10/25 (40%) of the values elsewhere in this paper.

Table 5: Components selected for adhesion testing

A (PLCC 68)	Texas
B (BGA 380)	
C (PLCC68)	EXAR
D (BGA177)	ANALOG
E (QFP 532)	
F (QFP 160)	Topline
G (BGA 256)	Amkor

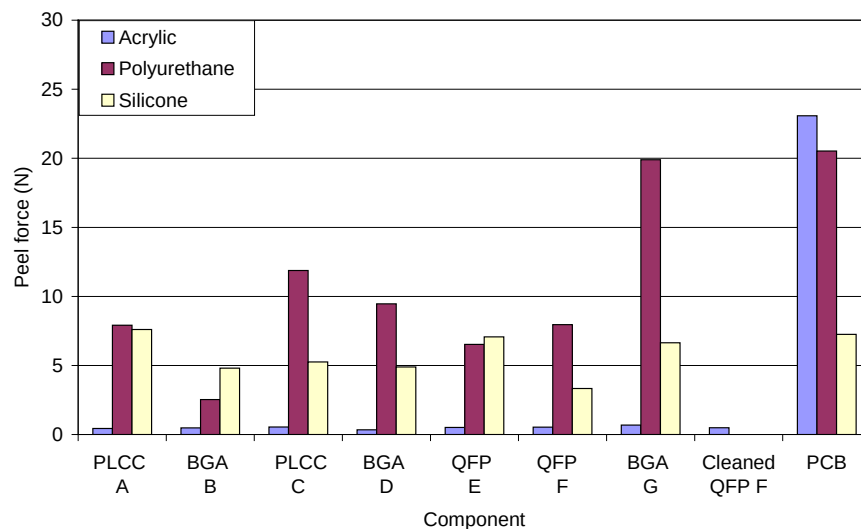


Figure 13: Average peel force from different components for three coatings

The results of the adhesion tests are presented in Figure 13, and the failure mode identified in Table 6.

Table 6: Failure mode from different components for three coatings
(T: coating/tape interface, S: coating/substrate interface)

Coating \ Component	A	B	C	D	E	F	G
Acrylic	S	S	S	S	S	S	S
Polyurethane	S	S	T	S	S	S	T
Silicone	T	S	T	S	T	S	T

The results of adhesion testing on components clearly show that for a wide range of components adhesion is problematic for coatings. The acrylic in this test struggled with all the components, the force not exceeding 1N. The polyurethane only achieved the optimum force on BGA G, with the force being significantly reduced for all the other components, particularly for BGA B. Even for the silicone coating, on three components the failure was at the substrate interface. Component QFP F was cleaned in IPA + DI water in an ultrasonic cleaning system for 20 minutes, and tested with the acrylic, but this had no effect on the adhesion. The low adhesion can be attributed to release agents in the plastic mould compound, and their lack of solubility in the coatings. Clearly components present a significant adhesion issue for coatings, and where the tape can be applied to a sufficient area, the technique can clearly differentiate between different coatings.

Post coating application of tape

The method described so far has the tape applied simultaneously with the coating. Here we explore the possibility adhering the tape by applying a second layer of the original coating. We refer to this as the so-called double coating method, shown schematically in Figure 14. If successful, this would obviate the need to always embed the tape in the initial coating, allowing any coating to be tested at any point in assembly without the need for any previous preparation. The experimental parameters of subsequent tests are shown in Table 7. Here we also explored the effect of state of cure of the first coat, it either being fully cured, or only cured for 1 hour.

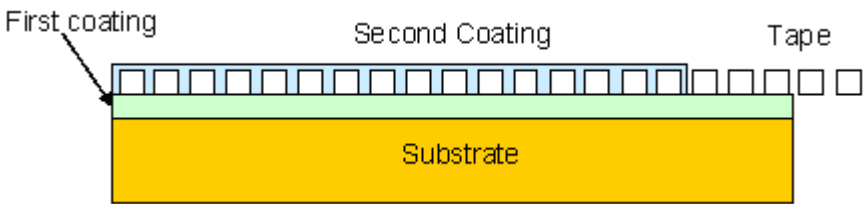


Figure 14: Double coating method

Table 7: Experiment details

Coating	Substrate	Cure of first coating
Acrylic B	Bare laminate	Fully cured
Polyurethane	Resist A	1 hour cured
Silicone		

Figure 15 shows the three possible failure modes as the tape is pulled from the substrate. Ideally the third failure mode should occur if useful information is to be gained on the adhesion of the original coat.

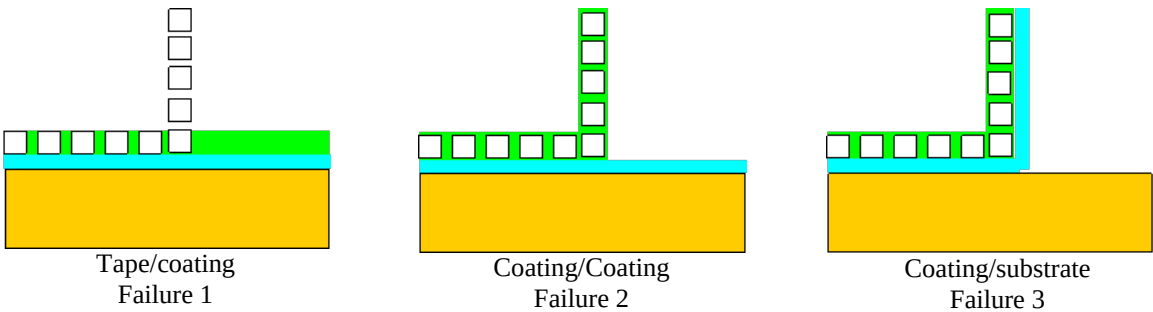


Figure 15: Three failure modes for the double coating method

Results for testing on coated bare laminate, and on a commercially available resist are presented in Figure 16. The testing of the double coating method showed poor adhesion of the first coating with resist samples and gave the desired type 3 failures for both the acrylic and polyurethane coatings. However, on the PCB higher adhesion forces for the acrylic on the PCB were observed, and the failure was type 1. This is disappointing as the second acrylic coat should dissolve the surface of the bottom coat and form a single coating. With the polyurethane type 3 failures were not observed, and low forces were recorded with the tape coming away too easily, type 1 and 2 failures. Low forces were also obtained with the silicone coating.

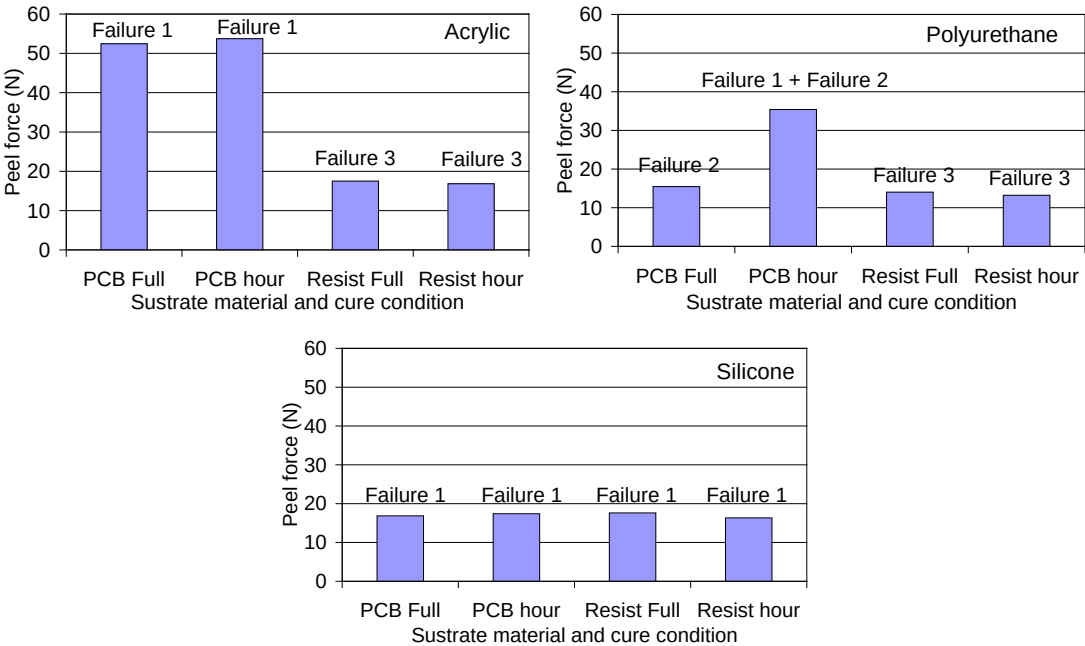


Figure 16: Peel force results for the three coatings with the double coating method.

Surface energy

The surface energy of a substrate will have an effect on adhesion, and dyne pens are used as an indicator of surface energy and hence adhesion. Here we attempted to measure the surface energy using deionised water, with an DAS 100 instrument , as shown in Figure 17. The DAS 100 dispensed a 70pl water drop for each measurement, and measured the wetting angles on both side of each drop, as presented in Figure 17, and took an average reading. Nine measurements were taken for each substrate coating combination. Wetting angle measurements were made on different substrate materials, bare laminate, copper clad laminate, and the resists and components identified above, and the results are given in Table 8.

The wetting angle measurements are compared with the adhesion values. The adhesion values are also given in Table 8, and have been normalised to a 10mm tape values, to enable comparison with the component tests. Unfortunately, a correlation was not established between these wetting angle measurements and the adhesion values measured in the peel test. For example, the acrylic testing on components gave universally low adhesion forces, but the wetting angle measurements are variable. The polyurethane coating on the BGA G gave a low wetting angle and high force, but QFP E had the same wetting angle but low force. It is possible that further work on selecting the test liquid may yield an improved correlation, for example selecting a liquid similar to that used in the dyne pens, rather than water. However, there is a potential difficulty in that hydrogen bonding formed during the coating cure cannot be directly predicted by a wetting angle measurement, as there is a chemical affinity element of this bonding which is independent of the surface energy. Neither can the solvent action of a coating be replicated by the dyne pens.

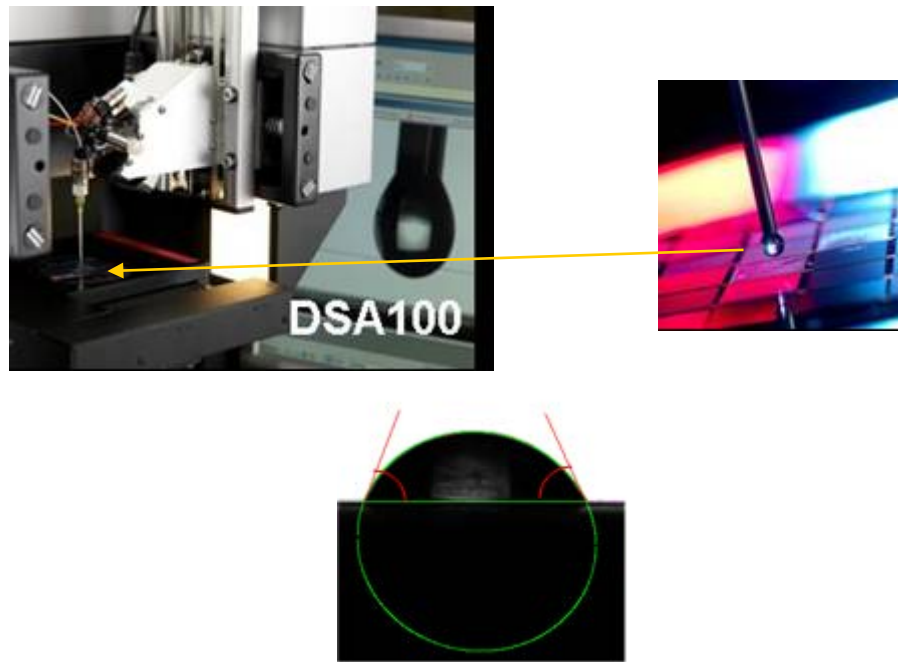


Figure 17: Wetting angle measurement with DSA 100

Table 8: Wetting angles of substrate and peel force from three coatings

Substrate material	Wetting angle (°)	Peel force (N/10mm)		
		Acrylic	Polyurethane	Silicone
FR4 PCB	70.0	21.8	19.7	6.7
Copper	86.9	20.9	12.1	6.9
Resist A (Curtain)	70.1	7.2	5.4	6.9
Resist A (Screen)	68.9	20.7	12.8	7.0
Resist B	71.0	20.7	16.4	6.9
Resist C	84.1	20.4	15.9	6.6
Resist D	72.3	20.1	17.1	7.1
PLCC A	91.3	0.4	7.9	7.2
BGA B	86.2	0.5	2.5	4.8
PLCC C	81.1	0.5	11.9	5.3
BGA D	84.6	0.3	9.5	4.9
QFP E	75.0	0.5	6.5	7.1
QFP F	92.9	0.5	8.0	3.3
BGA G	75.5	0.7	19.9	6.6

Conclusions

- We have demonstrated a adhesion peel test for conformal coatings that can detect when the adhesion becomes less than optimal for silicone, acrylic and polyurethane coatings.
- Selection of the tape material is critical to achieve the desired coating to substrate failure during peel. A high surface area woven fabric with a loose weave was found to be ideal, and a 65% polyester and 35% cotton mix cloth with 15% open area was selected.
- A limitation of the test is that the tape has to be applied with the coating, by fully immersing the tape in the coating and then curing.
- The peel test is not particularly sensitive to the variables of peel speed, peel length and tape width. It is envisaged that a wide range of peel test equipment could be utilised.

- Application of the test to a range of material variables was proved successful, and included the cure state of the coating, contamination of the substrate, surface energy state of resists and components. This test was extremely effective at revealing the low adhesion of coatings to plastic encapsulated IC packages. The test could be readily applied to coating process optimisation.
- Separation in the peel test was not always at the coating substrate interface, and both failure surfaces should be checked to identify the separation path. The silicone revealed low adhesion forces for removing the tape from the coating, a problem for the test, but on contaminated surfaces, the weaker coating substrate bond could be investigated. For the acrylic and polyurethane coatings, the force to remove the tape from the coating was significantly higher than for the silicone. The silicone had the least dependence on substrate surface condition.
- Where low adhesion occurs, the test peeled the coating from the substrate. Where the tape was pulled from the coating, it is indicative that the coating has sufficient adhesion for its functional purpose.

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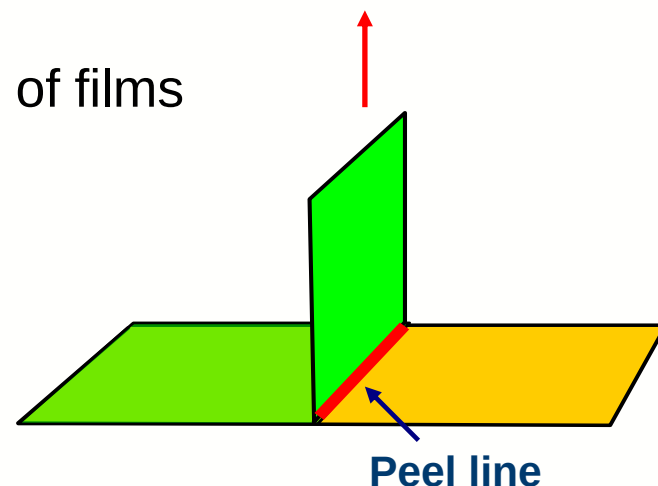
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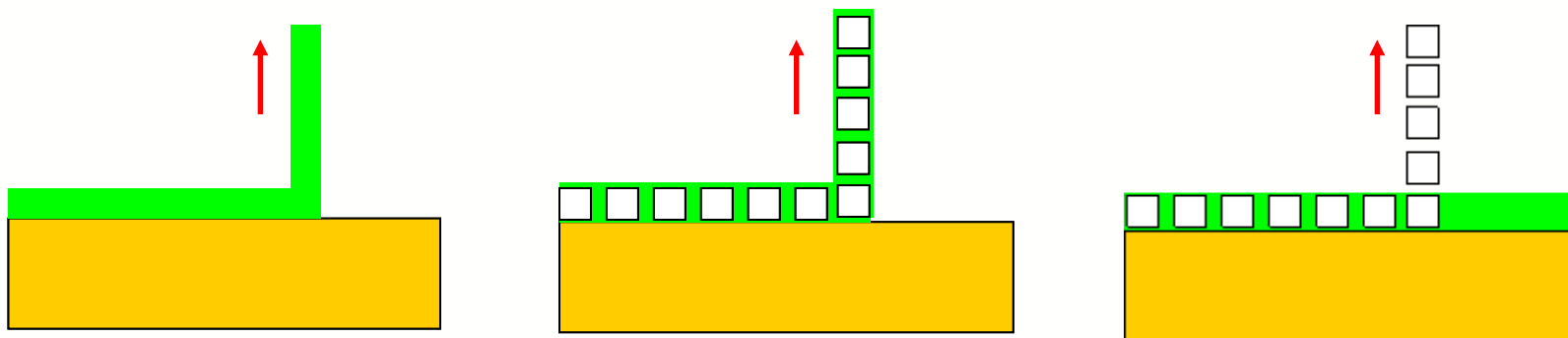


Introduction

- Conformal coatings are applied for circuit protection.
- They must act as a barrier and remain intact and well adhered to the surface.
- There is no quantitative method for coating adhesion measurements
- Any method should be easy to use and sensitive to different coatings and substrate surfaces.
- Peel testing is used for adhesion testing of films
- Cohesive strength must be higher than the adhesive strength



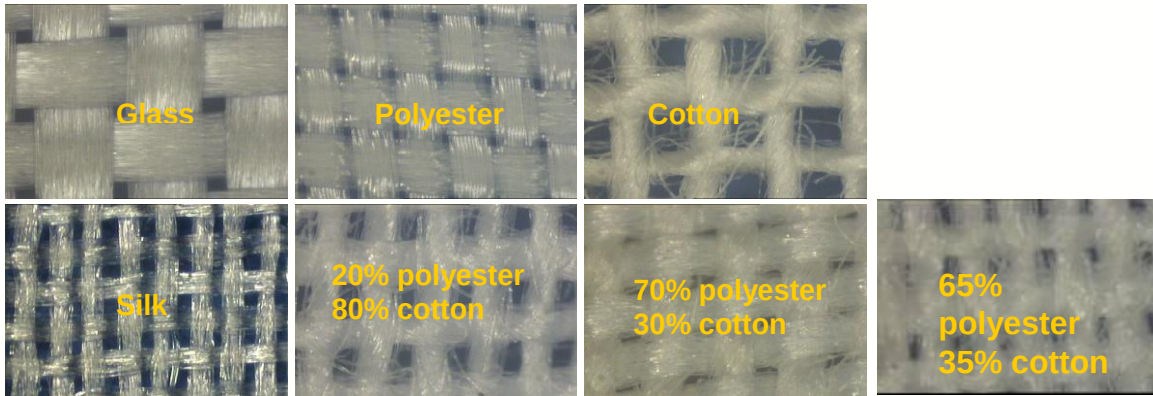
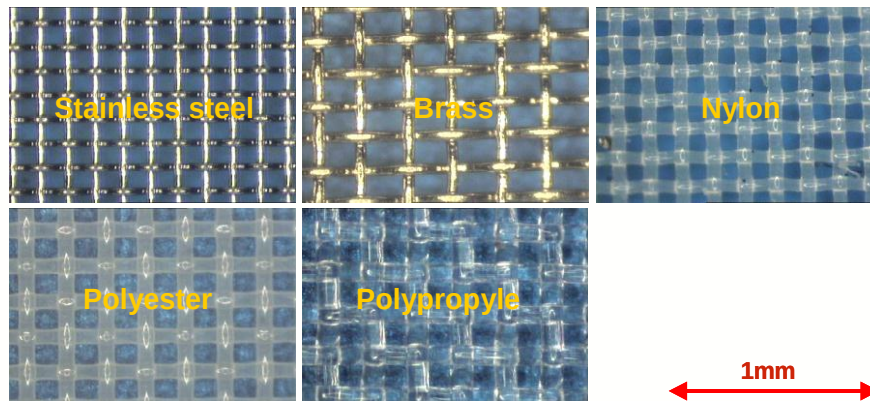
Challenge



- Coating adhesion force is higher than its cohesive strength, particularly for thin coating film ($<50\mu\text{m}$), direct peel on coating is impossible even with very thick coating.
- A flexible material is needed to reinforce the coating so that peel can be observed.
- Selection criteria for supporting material:
 - The material should have strong bond with coating.
 - Hence, test the peeling force between coating and substrate
 - Achieve discrimination between different levels of adhesion.
- The supporting material must not affect coating curing process.

Supporting materials ~ trials

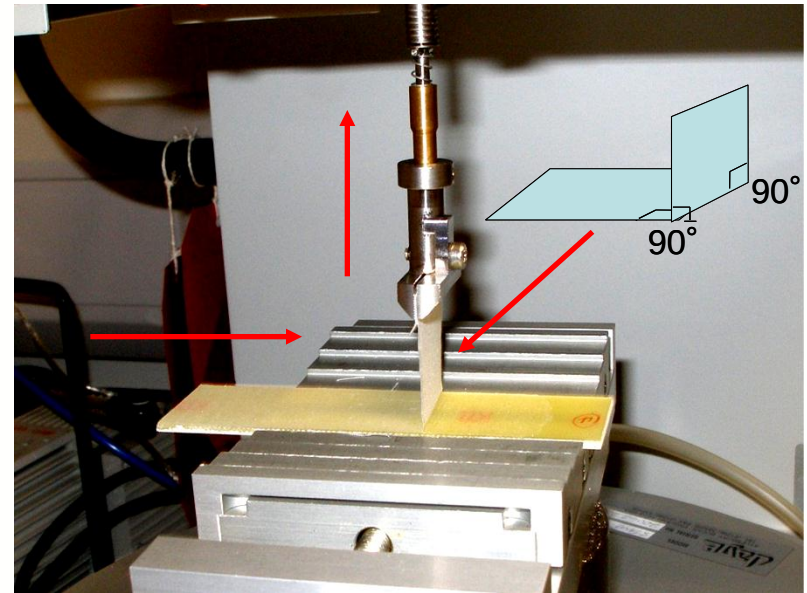
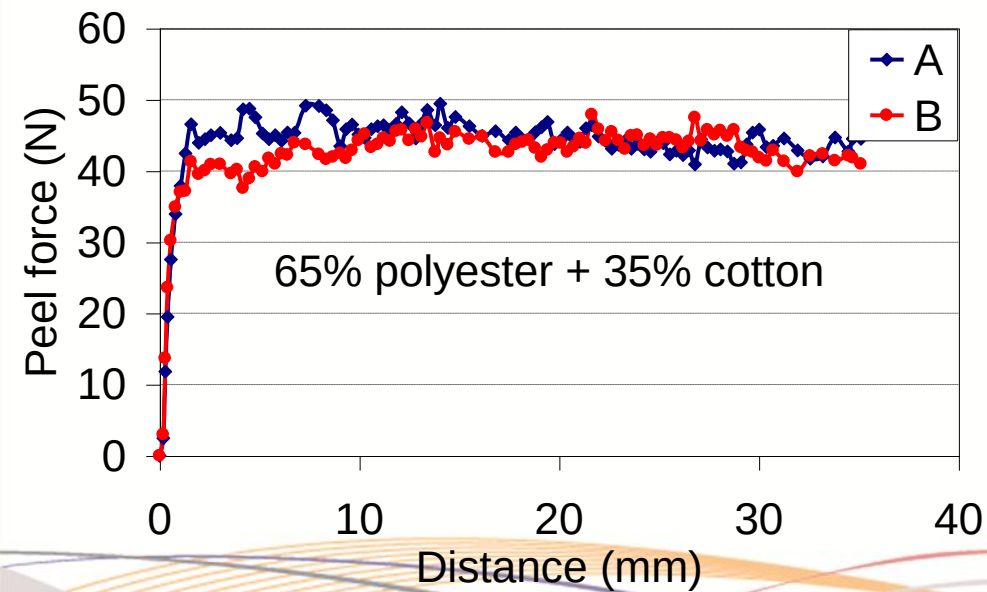
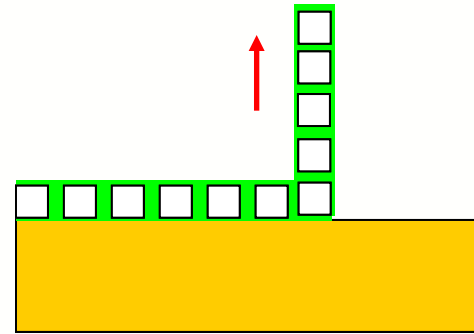
- Mesh and cloth are possible materials
- There is enough open area to allow the solvent in the coating to evaporate, and not affect the curing process.
- It is advantageous if the mesh and cloth have high surface areas. This will lead to increased adhesion area and increased adhesion force.



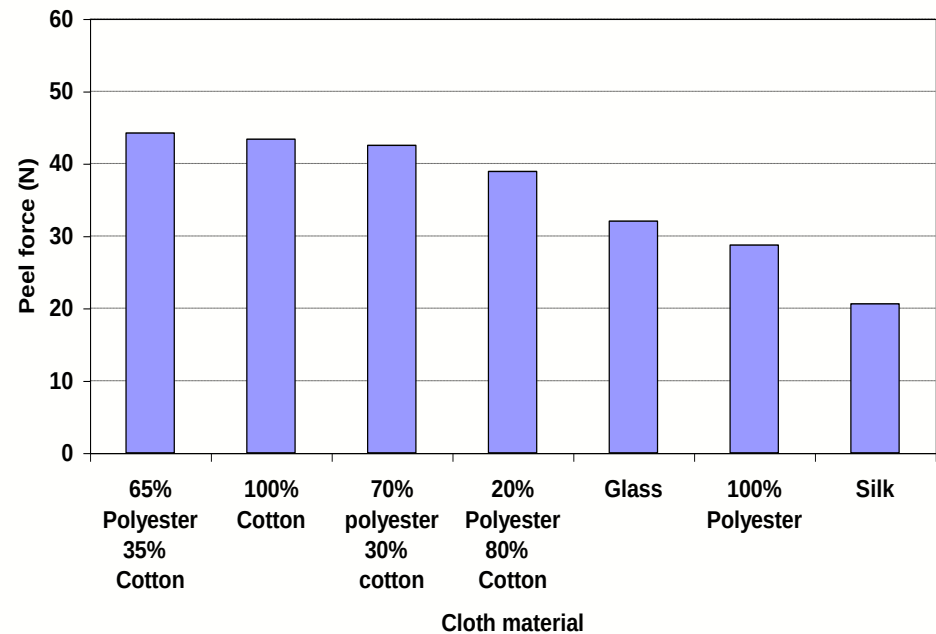
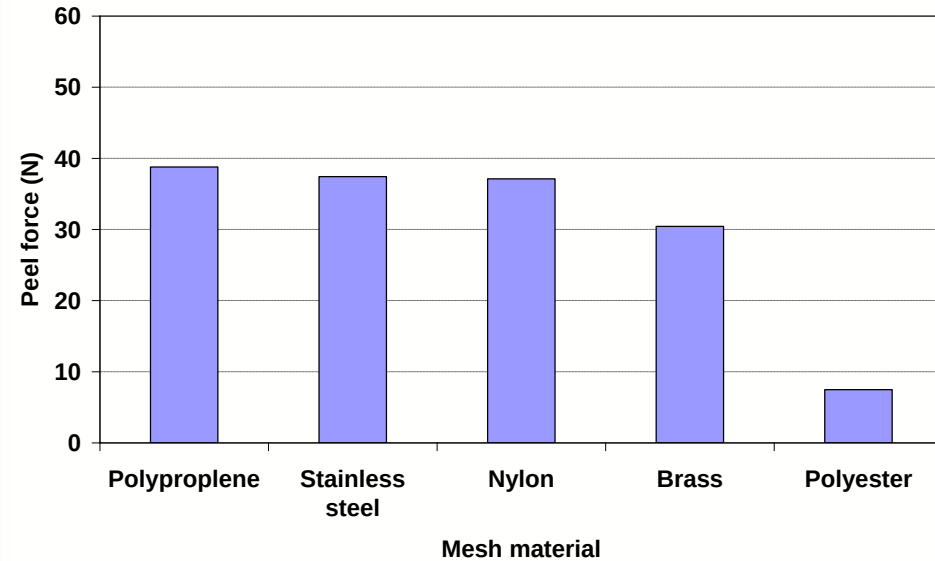
Evaluation of different supporting materials



- 25mm width sample-PCB
- Acrylic A coating
- 1mm/second peel speed

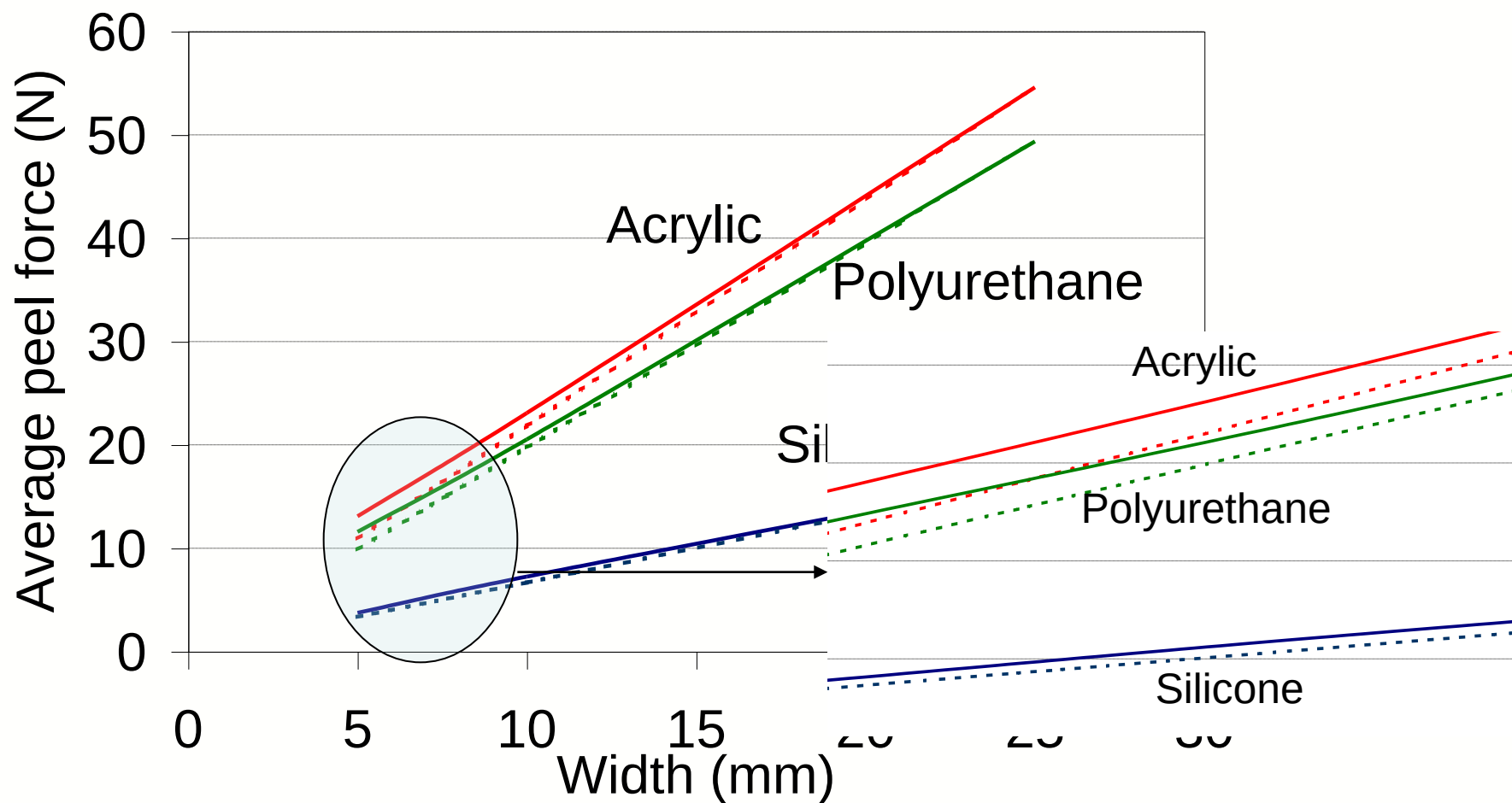


Peel force – mesh & cloth materials



- Peel force is dependent on materials and structure
- A single filament mesh does not show enough adhesion to the coatings
- Complex structure of cloths with high surface area improve coating adhesion.
- Selected the polyester/cotton 65/35%

Peel force with tape width



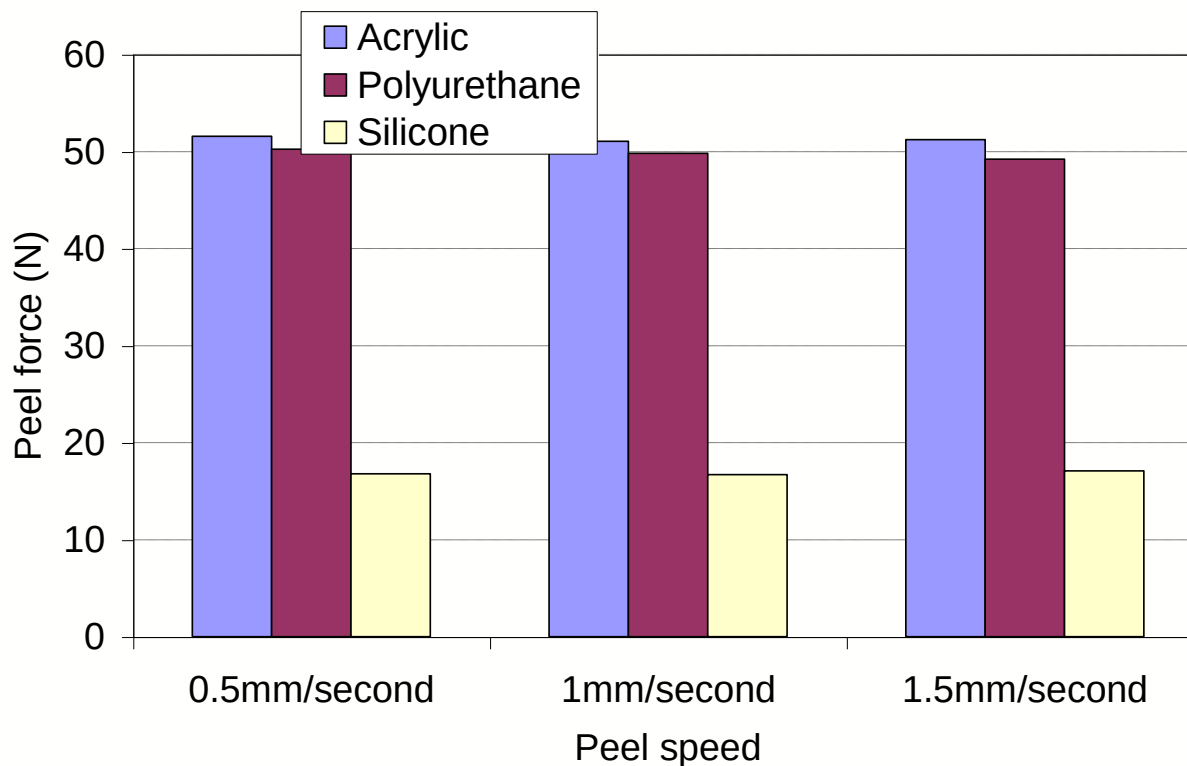
- Solid lines = measurement results, and dotted line is extrapolated from 25mm results

Peeling force versus width

- The peel force follows an approximate linear relationship with tape width, for all coatings.
- As the tape narrows, there is a discrepancy in the expected pro-rata peel force.
- This is probably because there is an edge effect. Apparently there are higher forces needed to separate the edge than the central areas of the tape.
- Therefore, it is recommended that not less than 10 mm tape be used to get accurate peel force result.

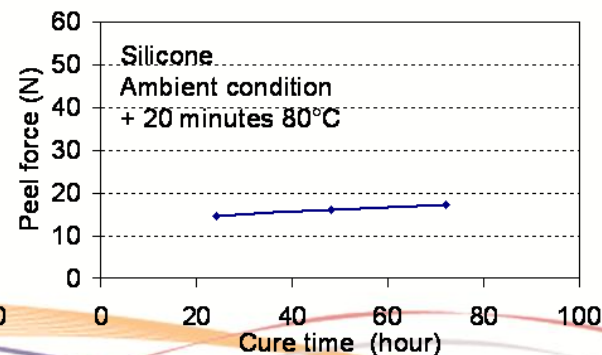
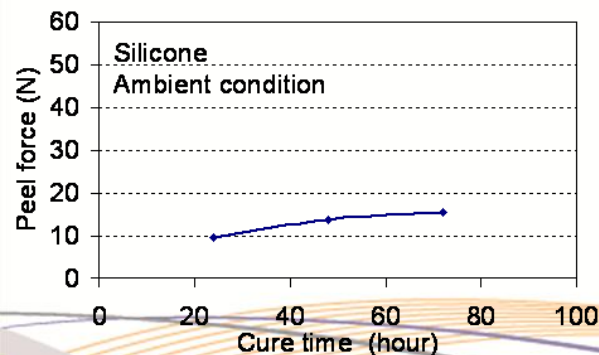
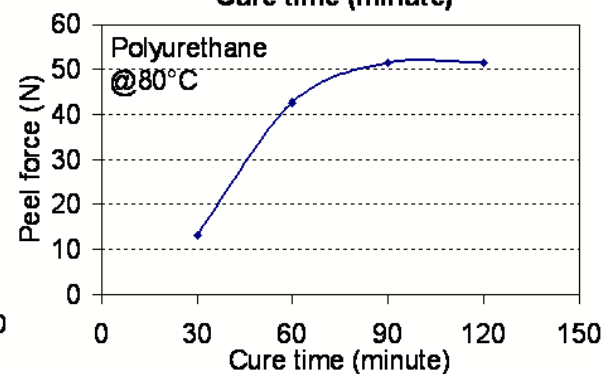
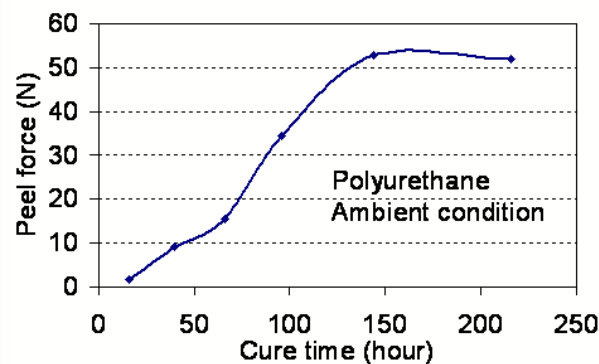
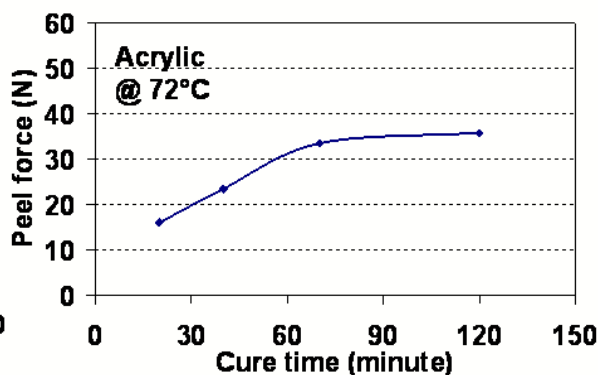
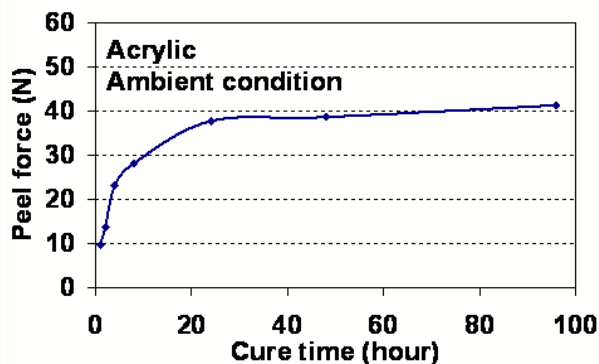


Effect of peel speed on peel force



- Coating adhesion force is not affected by peel speed in the tested range 0.5 to 2 mm/second, so any peel speed can be selected in this range.
- In our work 1mm/s was used

Cure of coatings

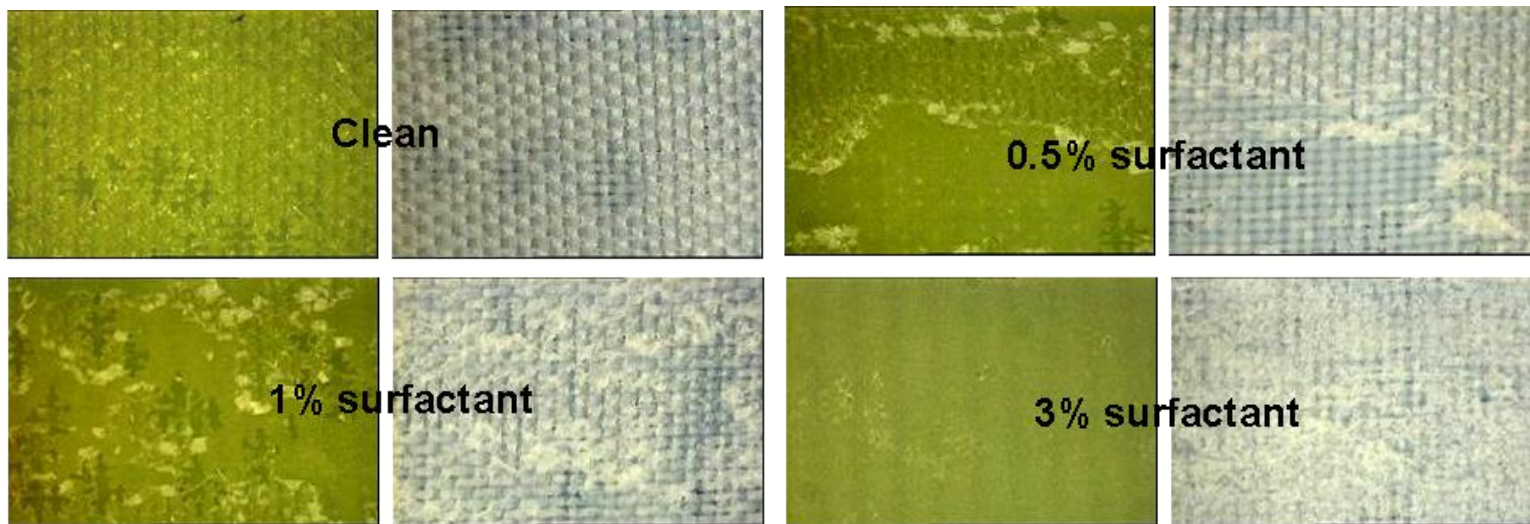


- At 72°C the acrylic bubbled.
- Future studies coatings cured for 24 hours at 22° C, plus
- Acrylic
 - 30min @ 80° C
- Polyurethane
 - 60min @ 80° C
- Silicone
 - 20min @ 80° C

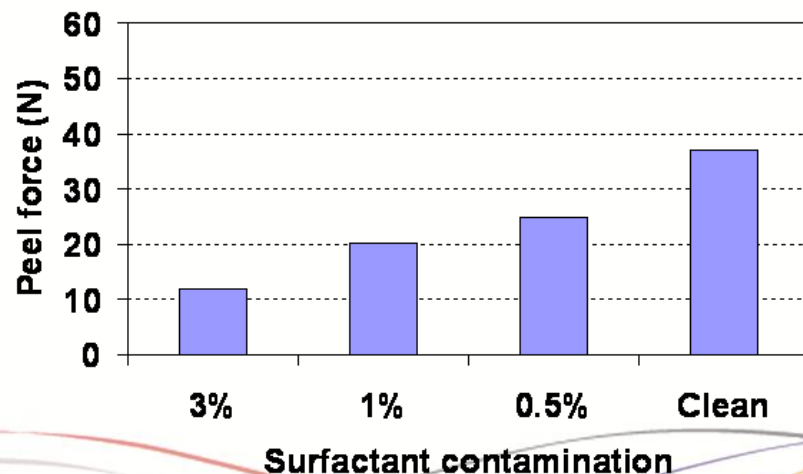
Contamination effects on adhesion with acrylic coating

- Clean PCB, failure always occurred at the tape/coating interface
- Establish test response as surface energy decreased
- Trialled a number of common products:
 - Solvent and water based solder fluxes
 - Various petroleum based greases with different melting points, a grease with added slip agents, silicone lubricants and a silicone release spray.
 - *All failed to impact significantly on the adhesion force*
 - Common surfactants were found to significantly impact adhesion.
 - Hence the laminate substrate was contaminated with a common proprietary surfactant, dissolved in DI water

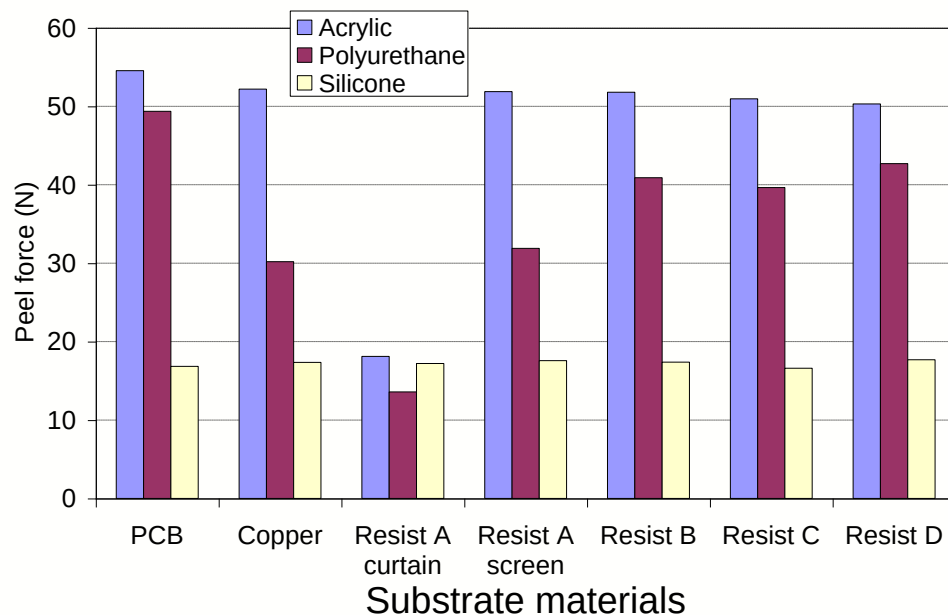
Surfactant concentration effect on adhesion



- With strong bonding failure occurred at coating/tape interface
- As the adhesive strength decreased failure moved to the coating/substrate interface
- Then the coating peel force can be measured directly.



Adhesion on solder resists and copper



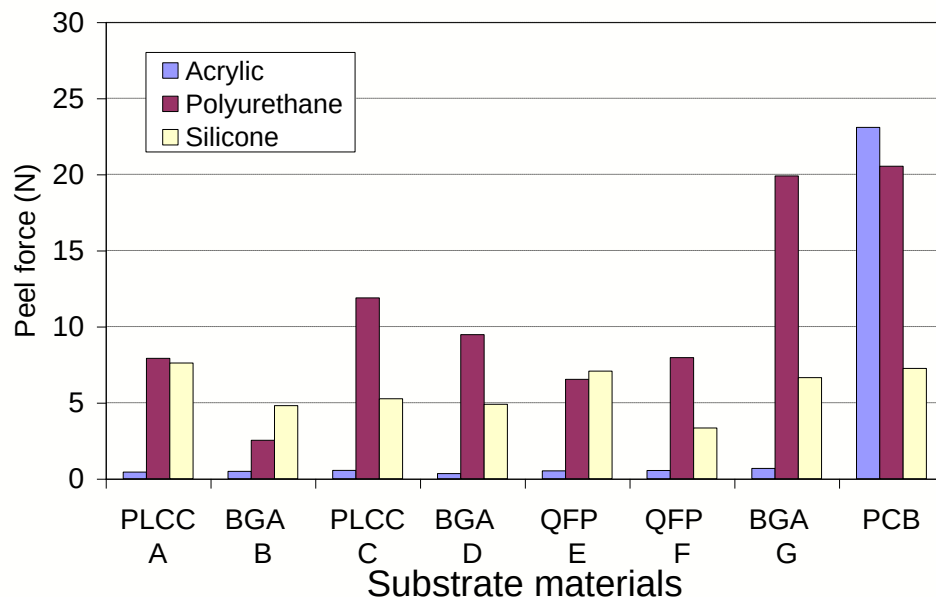
- Polyurethane is more sensitive to substrate
- Acrylic and polyurethane to whether Resist A is screen or curtain coated
- Silicones show low adhesion to tape, and no sensitivity to substrate
- All resists fully cured

Substrate Coating	Substrate		Resist				
	PCB	Cu	A Curtain	A Screen	B	C	D
Acrylic	T	T	S	T	T	T	T
Polyurethane	T	S	S	S	T+S	T+S	T+S
Silicone	T	T	T	T	T	T	T

- Coates XV501T-4
- Coates XV501 CAWN1287
- Huntsman Probimer 77
- Electra EMP100

* T: coating/tape interface S: coating/substrate interface

Coating adhesion to components



Component \ Coating	A	B	C	D	E	F	G
Acrylic	S	S	S	S	S	S	S
Polyurethane	T	S	S	S	S	S	S
Silicone	T	S	T	S	T	S	T

- T: coating/tape interface
- S: coating/substrate interface

- Component adhesion is problematic
- Acrylic struggled with components
- Polyurethane only achieved the optimum force on BGA G
- Silicone failed at the substrate interface for three components.
- Ultrasonic cleaning QFP F in IPA + DI water system for 20 minutes had no effect on adhesion
- Low adhesion can be attributed to release agents

A (PLCC 68) Texas

B (BGA 380) Texas

C (PLCC68) EXAR

D (BGA177) ANALOG

E (QFP 532) ANALOG

F (QFP 160) Topline

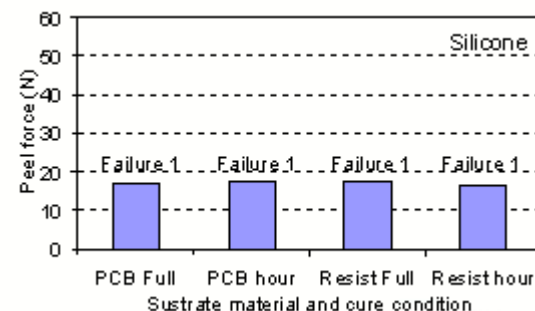
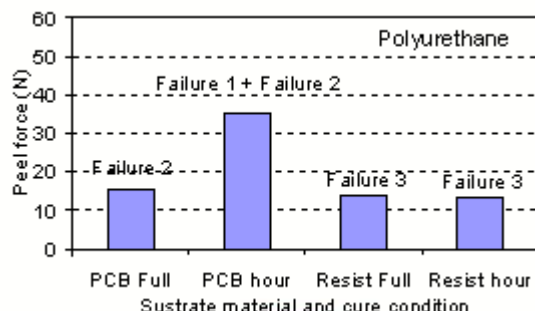
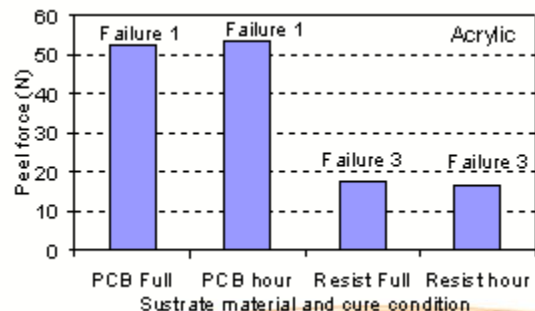
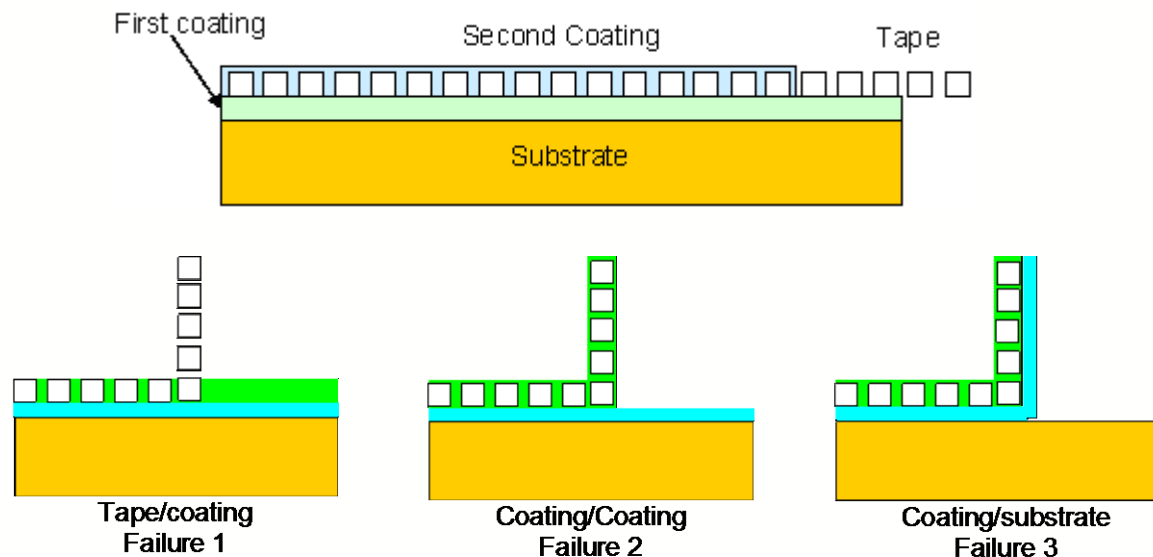
G (BGA 256) Amkor

Post coating application of tape

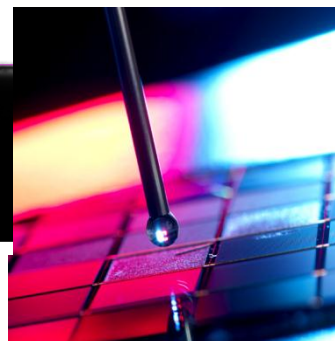
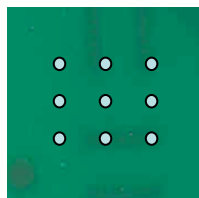
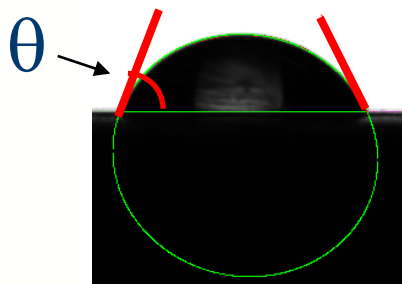
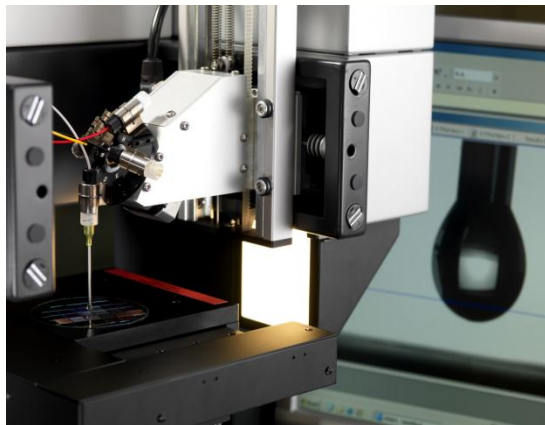
Coating	Substrate	Cure of first coating
Acrylic B Polyurethane Silicone	Bare laminate Resist A	Fully cured 1 hour cured

Double coating method

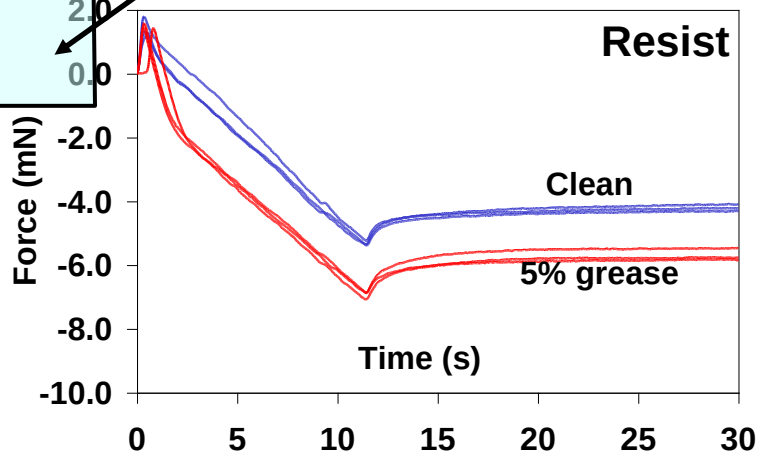
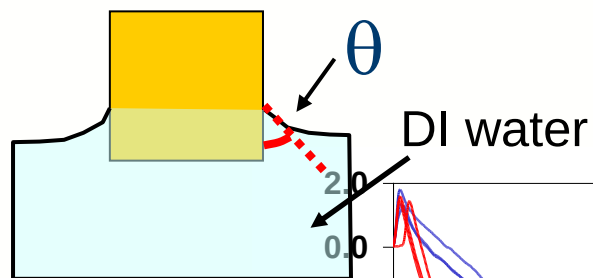
- Suitability of double coating method depends on coating chemistry
- Most success with polyurethane coating
- Mixed results with the acrylic, with low adhesion surfaces it appears to work
- Difficult to determine if this approach worked with the silicone coating



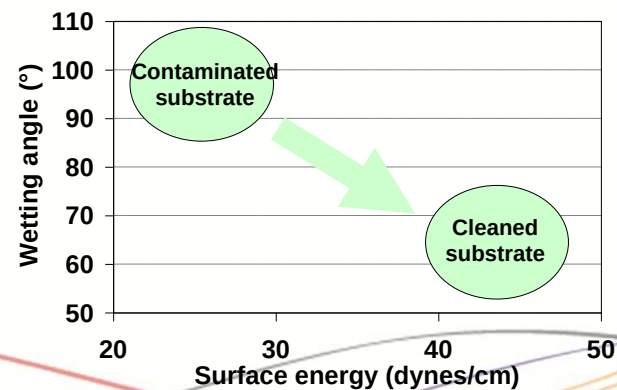
Surface energy measurements



- DI water
- 70pl drop
- Average of 9 measurements



Dyne pens



Conclusions

- Adhesion peel test for conformal coatings has been demonstrated
 - Detection of low adhesion for silicone, acrylic and polyurethane coatings.
 - Where low adhesion occurs, the test peeled the coating from the substrate
 - Where the tape was pulled from the coating, it is indicative that the coating has sufficient adhesion for its functional purpose.
- Selection of the tape material is critical to achieve the desired coating to substrate failure during peel.
 - A high surface area woven fabric was found to be ideal
- A limitation of the test is that the tape has to be applied with the coating
- The peel test is not particularly sensitive to test variables of peel speed, peel length and tape width. It is envisaged that a wide range of peel test equipment could be utilised.
- Application of the test to a range of material variables was proved successful, and included the cure state of the coating, contamination of the substrate, surface energy state of resists and components.
 - This test was extremely effective at revealing the low adhesion of coatings to plastic encapsulated IC packages.
 - The test could be readily applied to coating process optimisation.
- Separation in the peel test was not always at the coating substrate interface.

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 - This test was extremely effective at revealing the low adhesion of coatings to plastic encapsulated IC packages.
 - The test could be readily applied to coating process optimisation.
- Separation in the peel test was not always at the coating substrate interface, and both failure surfaces should be checked to identify the separation path.
 - The test did not work well universally with silicones
 - Acrylic and polyurethane coatings, the force was significantly higher