A PROTOCOL FOR DETERMINATION OF THE ADHESIVE FRACTURE TOUGHNESS OF FLEXIBLE LAMINATES BY PEEL TESTING: FIXED ARM AND T-PEEL METHODS

An ESIS Protocol
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1 INTRODUCTION.

Flexible laminates are used in a wide range of industrial and packaging applications. In general, there will be considerable practical importance associated with the adhesive strength between adjacent layers in these laminates. Sometimes it will be important to maximise this adhesive strength, sometimes it will be required to minimise it. Overall, the key requirement will be to control it and to achieve this it is first necessary to measure it. The purpose of this protocol is to provide guidance on the measurement of peel strength of the laminate and then to show how the adhesive fracture toughness (also known as adhesive strength or interfacial work of fracture) can be determined from this peel strength and other measurements.
Other standards provide guidance on conducting peel tests on flexible laminates (eg ISO 8510-1 1990 and ISO 8510-2 1990 “Peel test for a flexible-bonded-to-rigid specimen assembly, Part 1 90^o peel and Part 2 180^o peel”. ISO 11339 1993 “180^o peel test for flexible-to-flexible bonded assemblies (T-peel test)”). These procedures indicate how to measure peel strength (force per unit width for peeling) but do not include additional test information and analysis for converting peel strength to adhesive fracture toughness. There are important distinctions between these two properties in that peel strength indicates how difficult it is to peel one substrate from another, but adhesive fracture toughness is indicative of how well the two substrates are stuck together. It is this latter property that is most often sought.
The protocol is divided into two parts; one for ***a fixed arm peel test*** and the other for ***a T-peel test***. These geometries are different but there is a common aim in converting the peel strength measurement into adhesive fracture toughness. Some aspects of the analysis of the elastic and plastic deformations will be seen to be similar.
Laminates that can be peeled can be classified into two types. First, where the adhesive layer thickness is negligible e.g. two polymer films that are fused together. Second, where the adhesive layer thickness is not negligible and for which it might be necessary to accommodate the deformation within the adhesive layer during the peel bending process. Such laminates might include metal/polymer/metal systems. This protocol will accommodate both types of system.

2 FIXED ARM PEEL TEST.

2.1 ANALYSIS OF THE FIXED ARM PEEL TEST.

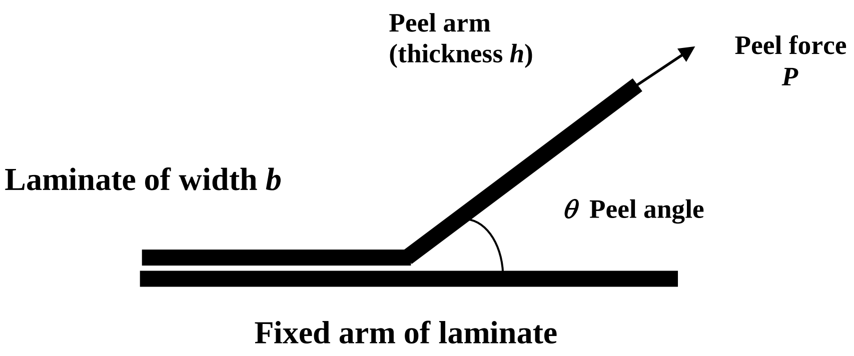


Figure 1 Fixed arm peel test

Figure 1 shows the peel of a laminate at a peel angle of θ with a force P acting on the peel arm (width b, thickness h). In order to peel one layer of the laminate from the other, it is necessary to provide energy in the form of external work to the laminate.
The energy contributions to a fixed arm peel procedure can be described by a global energy analysis [1]. The input energy to the peel test needs to be resolved into the various deformational energies, elastic, plastic and adhesive fracture energies, at least.

$$G_A = \frac{dU_{ext}}{bda} - \frac{dU_s}{bda} - \frac{dU_{dt}}{bda} - \frac{dU_{db}}{bda} \qquad (1)$$

where G_A is the adhesive fracture toughness, U is energy with subscripts *ext*, *s*, *dt* and *db* referring to external work, strain energy, dissipated tensile energy and dissipated bending energy, respectively. b is the specimen width and da the peel fracture length. This approach has been applied to a fixed arm peel test [1, 2] in order to convert peel strength (P/b) to adhesive fracture toughness:

$$\boldsymbol{G_A = G - G_P} \qquad (2)$$

G is the input energy after correction for tensile elastic and plastic deformation in the peel arm and G_P is the plastic work in bending the peel arm. The tensile corrections are often negligible [1] and then input energy is given by:

$$\boldsymbol{G = \frac{P}{b}(1 - \cos\theta)} \qquad (3)$$

θ is the peel angle. In order to calculate the plastic deformation energy associated with the peel arm, it is first necessary to have knowledge of the tensile stress-strain characteristics of the peel arm material. This will include an initial elastic deformation but also a subsequent work hardening, plastic, deformation. The plastic bending in the peel tests may then be modelled using large-displacement beam theory with modifications for plastic bending. Solutions have been formulated for three cases:

(i) **Digitised**, where the stress-strain behaviour of the peel arm is digitised and a numerical analysis is used to calculate G_P [3].

(ii) **Bilinear,** where a bilinear fit is given to the stress-strain curve and an analytical calculation of G_P [4,5].

(iii) **Power law**, where an linear elastic-power law plastic fit is given to the stress-strain curve and an analytical calculation of G_P [4,5].

For the bilinear and power law curve fits, the details are as follows where a number of parameters from the stress-strain fits are calculated. N is a constant, α is the ratio of plastic modulus to elastic modulus i.e. E_2/E_1, σ_y is yield stress and ε_y is yield strain. N and α are the respective work hardening coefficients for power law and linear work hardening.

When $\varepsilon \leq \varepsilon_y$,

$$\sigma = E\varepsilon \tag{3}$$

in both cases, and when $\varepsilon > \varepsilon_y$,

$$\sigma = \sigma_y \left(\frac{\varepsilon}{\varepsilon_y}\right)^N \tag{4}$$

in the power law work hardening model, and

$$\sigma = \sigma_y + \alpha E(\varepsilon - \varepsilon_y) \tag{5}$$

for the bilinear model.

For laminates where the adhesive layer thickness (h_A) is very small, $h_A \to 0$, there is no requirement to consider the deformation in the adhesive in conducting the calculations of adhesive fracture toughness [4]. However, when $h_A > 0$ the deformation in the adhesive layer should be included in the analysis for which it will be necessary to have knowledge of the modulus of the adhesive material (E_A). In all, these various calculations can be complex and whilst a theoretical analysis is given in reference [4], software *(ICPeel)* that can be used to conduct the calculations is available on the Imperial College London website [6].

The digitisation of the stress-strain behaviour also requires a software package as does the subsequent numerical analysis for calculation of G_P. This is also provided on the Imperial College London website [6] where the package is called *ICPeel (Digitised)*.

In principle, the digitised approach should be the most accurate but it is also the most cumbersome and time-consuming approach. However, the digitised package is quite quick if an approximate solution is used first. This is based on using the stress-strain parameters form the curve fitting procedures prior to conducting an accurate analysis. Therefore, it is necessary to run *ICPeel* first and *ICPeel (Digitised)* next. Consequently, it is helpful to use the results from all three cases in the determination of G_P and G_A. The website includes instructions on how to run the packages.

In order to determine G_A without neglecting any of the elastic or plastic deformations, two experiments are required:

(a) The peel test with a control of the peel angle.

(b) A tensile stress-strain measurement of the peel arm up to fracture or at least to a strain magnitude that exceeds the maximum strain in the peel test (this is given in *ICPeel* and is typically 6%).

Experimental requirements for both of these measurements are given in section 2.2 and 3.2.

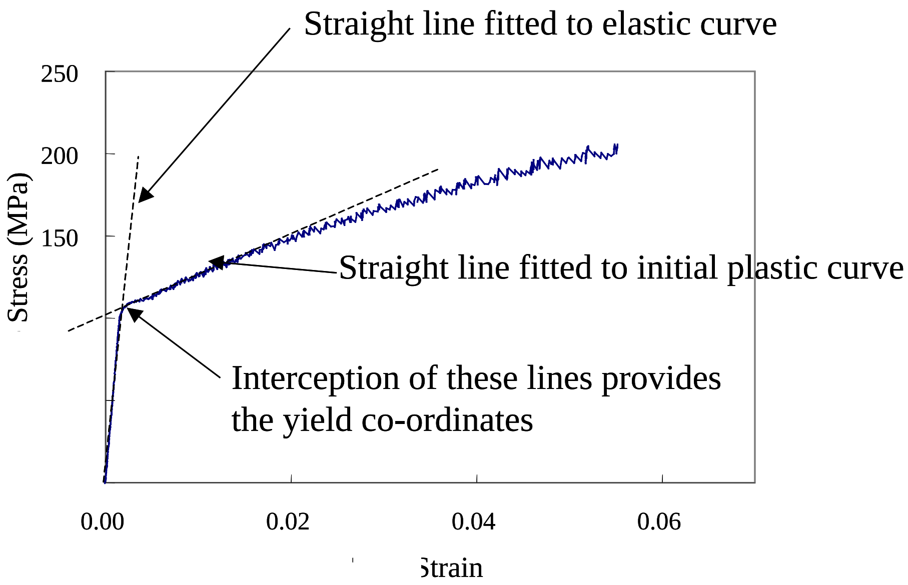


Figure 2 Definition of yield co-ordinates

A critical issue in analysing these curves concerns the definition of the yield co-ordinates (ε_y, σ_y). These are obtained from the experimental data by fitting a straight line to the linear elastic region of the curve and a straight line to the initial plastic region of the curve, as shown in Figure 2. The straight line fitted to the initial plastic curve should start beyond an approximate yield point and end at approximately 10 times the yield strain, again as shown in Figure 2. The interception of these two lines defines the yield co-ordinates.

The measured stress-strain curve is then modelled to either a bilinear form, as shown in Figure 3 or a power law form as shown in Figure 4. Both fits should be conducted for the purpose of calculation of G_P and G_A, but also for the *ICPeel (Digitised)* analysis. The fitted curves must comply with these yield co-ordinates (as equations 3-5 indicate). Therefore, once the yield co-ordinates are defined from the experimental curve, then the linear elastic portion for both models is the line between the origin and these co-ordinates. The plastic region then starts beyond the yield co-ordinates but for both types of model, the fitted curve must pass through the yield co-ordinates.

In general, net stress (force/original cross sectional area) and net strain (increase in length/gauge length) are used. However, if the strain exceeds 10% then a true stress should be used (true stress = net stress [1+ strain]).

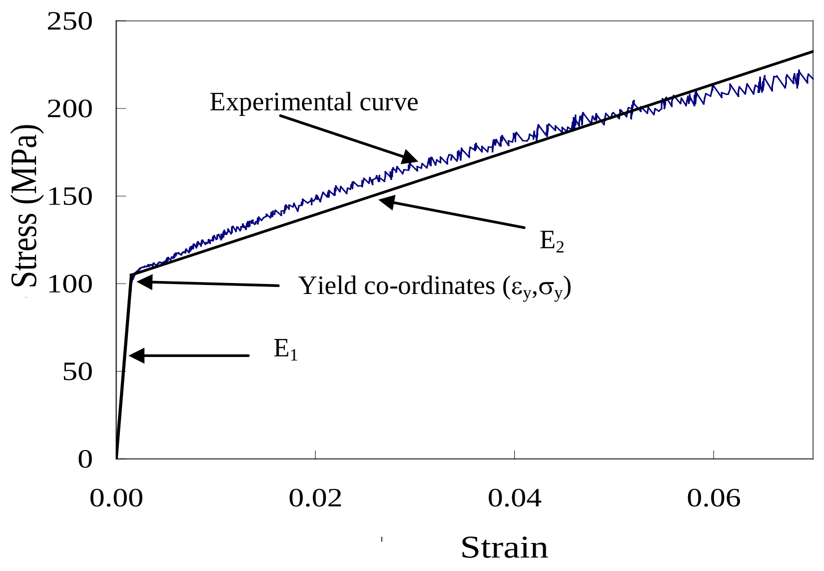


Figure 3 Tensile stress versus strain plot for a peel arm illustrating a bilinear elastic-plastic fit and the definition of E_1, E_2 and yield strain

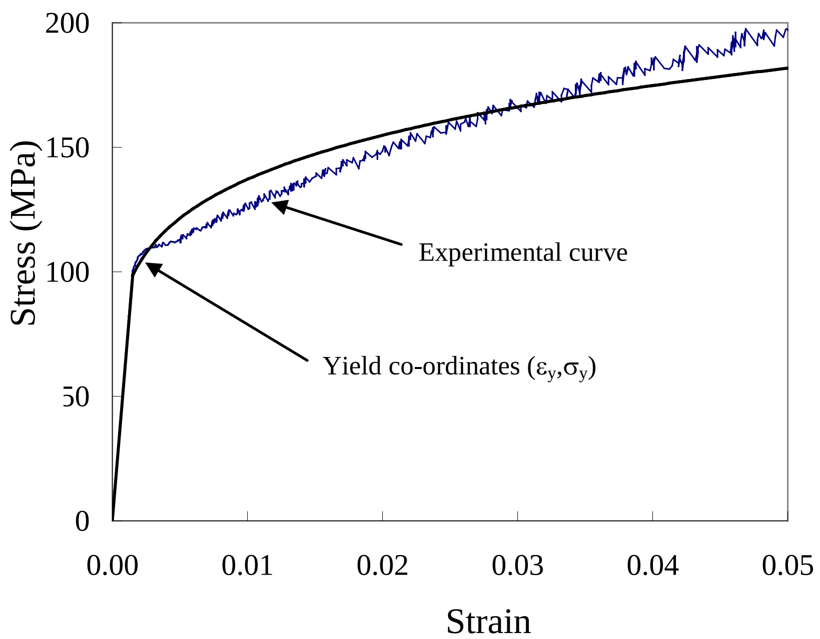


Figure 4 An illustration of an elastic region followed by a power law fit for work hardening.

Adhesive fracture toughness is determined according to equation 2. Ideally, the corrections for plastic deformation should not be too large otherwise errors for the determination of

adhesive fracture toughness will become significant. The size of this correction is given by $\left(\frac{G_P}{G}\right)x100\%$; the smaller this correction the better [2].
These considerations apply equally to the T-peel analysis but can only be determined for each peel arm separately.

2.2 EXPERIMENTAL PROCEDURES IN THE FIXED ARM PEEL TEST.

Specimens for conducting peel strength should be in the form of rectangular specimens where the two parts of the laminate have already been adhered but where there is a region of unadhered material (of nominal length 30 mm). The overall dimensions of a peel specimen need not be rigidly defined but for many tests we have found that a length of 100 mm and width 20 mm proves to be quite satisfactory. Three specimens should be tested for each set of conditions.
The choice of peel jig is not unique but the apparatus should incorporate a number of facilities. A successful kit is shown schematically in Figure 5. First, the apparatus should be able to select the peel angle in the range up to 180°. Second, the jig is attached to an Instron or similar universal testing machine such that as peel occurs the peel angle is maintained constant by the jig moving along a low friction linear bearing system. Third, only one side of the laminate is allowed to be the peel arm in the test. Of course, the laminate can be reversed so changing the peel arm material in another but separate test. (Consequently, the composition of the adherends may or may not be the same). Adhering one side of the laminate to the peel table is a critical issue. If this layer can separate from the table during the test then the energy involved in that process will increase the measured adhesive fracture toughness value to an erroneously high level. The means of gluing the layer to the table should be reported.
The material used for the peel arm (substrate) should be reported, as should the length, width and thickness dimensions of the specimen (h and h_A where h is the thickness of the substrate alone (i.e. without any coating of adhesive) and h_A is the thickness of the adhesive).
The peel angle needs to be selected on the basis of ensuring that the peel force is large compared with the resolution of the load cell and the frictional forces in the test. For purposes of the protocol it will be necessary to conduct the test at a range of peel angles between 45^{o} and 135^{o} but the peel angle 90^{o} should always be one of the angles. A peel test speed $\left(\frac{\Delta\delta}{\Delta t}\right)$ of 10 mm/min can be used as a standard speed with the 90^{o}. However, the peel angle will influence the peel crack speed $\left(\frac{\Delta a}{\Delta t}\right)$.

$$\frac{\Delta\delta}{\Delta t} = \frac{\Delta a}{\Delta t}(1-\cos\theta) \qquad (6)$$

Therefore, when conducting tests over a range of peel angles it us necessary to select a common peel crack speed.

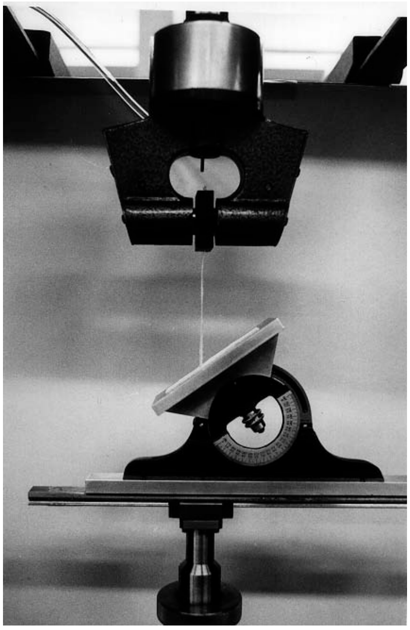


Figure 5 Fixed arm peel fixture with linear bearing system showing a peel angle of 45^{o}.

The force versus displacement curve to initiate and propagate a peel fracture should be recorded. At least 30 mm of peel fracture should be established. The average peel force should then be determined as shown in Figure 6A. If there is a combination of adhesive and cohesive fracture or “stick-slip” (as illustrated in Figure 6B), then for these occasions, both the mean lowest and the mean highest steady propagation peel force values should then be used to determine the adhesive and cohesive fracture toughness values, respectively.

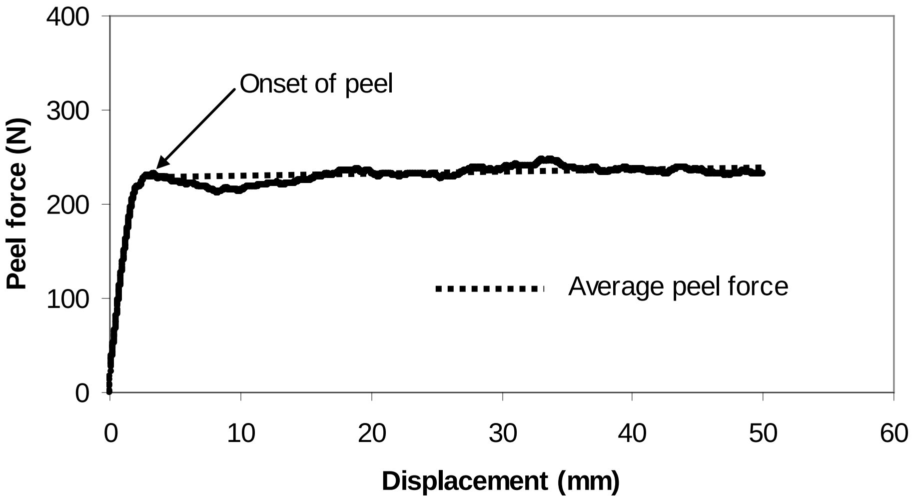


Figure 6A: Illustrative peel force versus displacement demonstrating how to average the peel force.

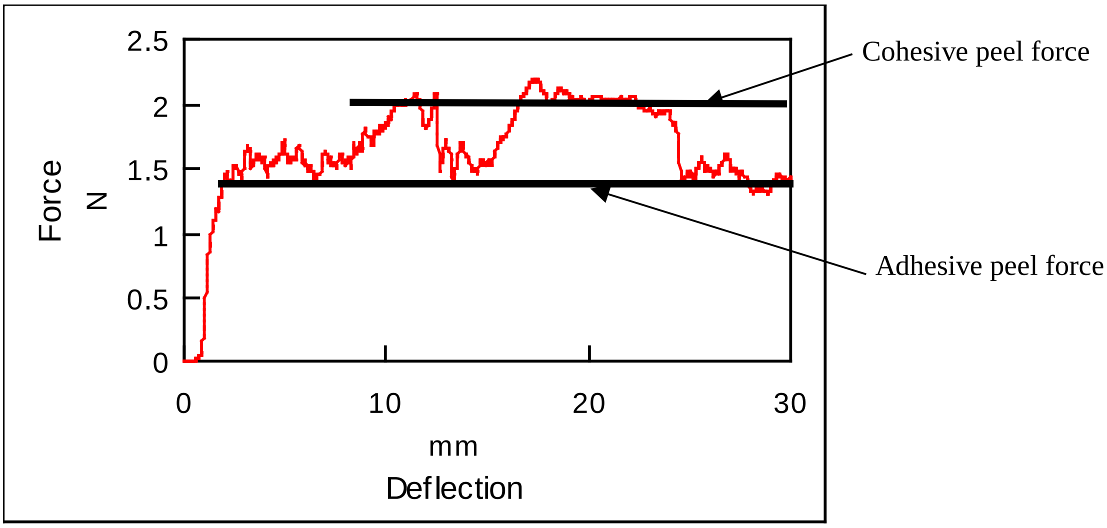


Figure 6B Peel force versus deflection in a fixed arm peel test. The higher peel forces relate to cohesive fracture; the lower peel forces relate to adhesive fracture

In order to conduct the corrections summarised in equations 2-5 it is necessary to obtain a tensile stress - strain plot on the material of the peel arm. (This should be the substrate material alone i.e. it should not be the substrate with a coating of adhesive material). In order to accommodate current analytical methods for the determination of the plastic work in bending the peel arm, this stress-strain plot will need to be described either as a bilinear function or as a linear elastic-power law plastic function or as a digitised function.

The tensile test should be conducted at the same test speed as the peel test and in order to obtain sufficient accuracy the test specimen should be a rectangular strip of width 10 mm and length 100 mm. In addition, an extensometer will be required to measure the strain deformations necessary to define the elastic deformations. The extensometer, ideally, should be of a non-contacting type, since the peel arms are likely to be of low stiffness. The tensile test should continue in order to enable a clear definition of the plastic region of the deformations by continuing the test to fracture.

The comments preceding Figure 3 provide instructions for fitting a bilinear elastic-plastic fit for the stress-strain curve. Five parameters should be obtained, namely E_1, E_2, α (E_2/E_1), εy and σ_y. The elastic modulus (E_1) is defined as the slope of the stress-strain curve at small deformations and before yielding has occurred. In order to define the plastic modulus (E_2) it is helpful to allow the tensile specimen to continue extension for as long as possible. If it transpires that the "work hardening" portion of the stress-strain curve exhibits a negative slope, then values of α and E_2 should be made zero (and not negative).

In order to construct a linear elastic region followed by a power law work hardening region, the scheme shown in the text preceding Figure 4 should be used. The definition of the elastic region is as above and the power law fit should only be made for post yield data. The parameters that should be obtained from the analysis are E_1, N, εy and σ_y. The co-ordinates at yield (as defined in an earlier section) are given by the intercept of the elastic line and the plastic region for the experimental curve. Therefore, if the general power law gives

$$\sigma = A\varepsilon^N \qquad (7)$$

The elastic region is

$$\sigma = E_1 \varepsilon \quad (8)$$

At yield, the stress (σ) is σ_y and the strain (ε) is ε_y. Therefore eliminating σ_y from equations 7 and 8 gives an expression for yield strain,

$$\boldsymbol{\varepsilon_y = (\frac{A}{E_1})^{\frac{1}{1-N}}} \quad (9)$$

and yield stress is

$$\sigma_y = E_1 \varepsilon_y \quad (9)$$

2.3 TEST REPORT FOR THE FIXED ARM PEEL TESTS

The following information is required in the test report.

General information
Name of Laboratory
Description of laminate
Material of peel arm
Adhesive used for gluing one arm to the peel table
Test equipment

Data from tensile test on the peel arm material
Test speed (mm/min)

Bilinear fit	**Power law fit**
Low strain modulus (E_1) (GPa)	Low strain modulus (E_1) (GPa)
Yield strain (%)	N
High strain modulus (E_2) (GPa)	Yield strain (%)
Alpha (E_2/E_1)	Yield stress (MPa)
Yield stress (MPa)	

Data from the peel test
Peel angle (°)
Test speed (mm/min)
Specimen dimensions L (mm)
b (mm)
Peel arm thickness h (μm) (substrate only)
Thickness of adhesive layer h_A (μm)
(if $h_A > 0$ then conduct calculations for two cases $h_A = 0$ and the actual value for h_A)
Value ascribed to the modulus of the adhesive E_A (GPa)
Peel strength (P/b) (N/mm)

Derived results by calculations (for each of the three fits to the stress-strain data)
G (J/m^2)
G_P (J/m^2)

G_A (J/m^2)
Correction factor (%)

Results should be presented for each specimen.

The report should include a plot of the peel curve (i.e. force versus displacement in the peel test). The length of peel growth should be marked on this curve together with a clear indication as to how the peel force used to determine the peel strength is derived from the plot. (Figure 6 shows an illustration of the requirement**).** In addition, the tensile stress-strain plots with the bilinear and/or power law fits should be reported

3 T-PEEL TEST

3.1 ANALYSIS OF THE T-PEEL TEST.

Figure 7 shows the specimen configuration during a T-peel test. When the stiffness of the peel arms is different the peel angles will be ϕ and θ (rather than 90^{o}). In Figure 7 the stiffer arm is peel arm 2, therefore ϕ<90 and θ>90. The analysis proceeds along similar lines to that in the fixed arm peel test (see section 2.1), except that there are now two peel arms to accommodate. However, only one of the peel angles needs to be considered since $\phi = \pi - \theta$:-

$$\boldsymbol{G_1 = \frac{P}{b}(1+\cos\phi)} \tag{6}$$

$$\boldsymbol{G_2 = \frac{P}{b}(1-\cos\phi)} \tag{7}$$

Where the super scripts *1* and *2* refer to the two peel arms, respectively.

The peel toughness terms for elastic corrections are then similar to those in equation (3), except that there are two terms. In a similar manner, there will be two terms for the dissipated energy. Consequently, there will be two forms for equations (2):-

$$\boldsymbol{(G_A)_1 = G_1 - (G_P)_1} \tag{8}$$

$$\boldsymbol{(G_A)_2 = G_2 - (G_P)_2} \tag{9}$$

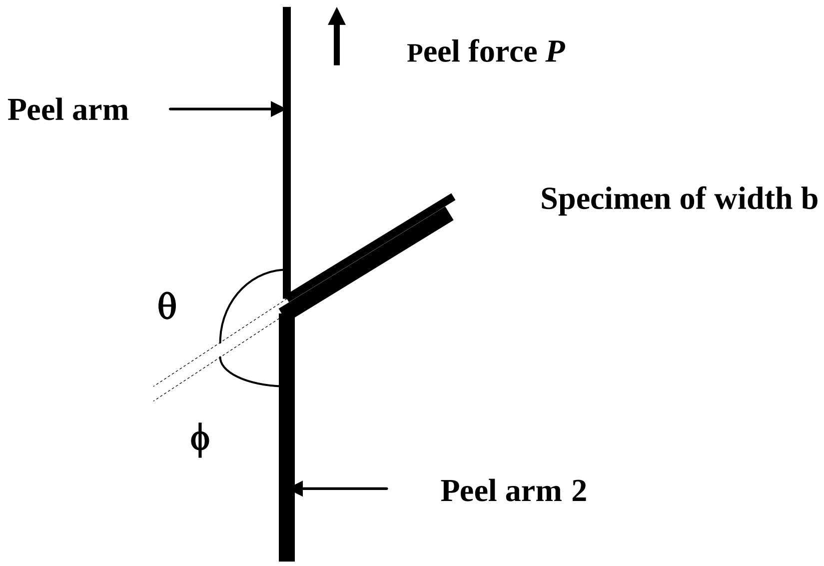


Figure 7: T-Peel Specimen During a Test.

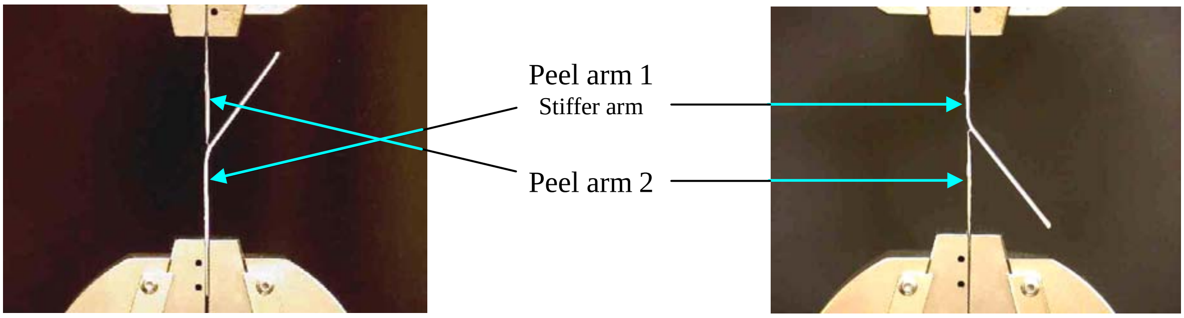
The adhesive fracture toughness (G_A) from the T-peel test is then the sum of the terms from equations (8) and (9), namely:-

$$G_A = (G_A)_1 + (G_A)_2 \tag{10}$$

3.2 EXPERIMENTAL PROCEDURES IN THE T- PEEL TEST.

Specimens for conducting peel strength should be in the form of rectangular specimens where the two parts of the laminate have already been adhered but where there is a region of unadhered material (of nominal length 30 mm). The overall dimensions of a peel specimen need not be rigidly defined but for many tests we have found that a length of 100 mm and width 20 mm proves to be quite satisfactory. Three specimens should be tested for each set of conditions and care should be taken to ensure that no part of the peel arms can touch the test equipment.

There are two configurations for conducting the T-peel tests, as shown in Figure 8. Configuration A has the stiffer peel arm at the bottom, whilst configuration B has the stiffer peel arm at the top. It is likely that the small peel angle for these configurations will not be the same since the stiffer peel arm, which could contain more mass than the less stiff peel arm, will hang differently. Three specimens tested in either configuration A or B should be conducted and the configuration type recorded.



Configuration A   Configuration B

Figure 8 Specimen configurations for T-peel

The materials of the peel arms may or may not be the same. The test machine should have the usual capabilities for sufficient resolution of force. Once the peel arms are in tension, the specimen configuration will be similar to one of those shown in Figure 8. During the course of peeling it is necessary to measure one of the peel angles (ϕ or φ) and at least three measurements should be made throughout the 30 mm peel fracture; one near the start, one in the middle and one near the end. The average value for the peel angle can then be determined and used in the calculations.

A peel test speed of 10 mm/min can be used as a standard. However, if there are large variations in measured peel angle (e.g. > 30^{o}) between specimens or samples then equation 6 should be used to adjust to a common crack speed. (This will be a retrospective judgement). The force versus displacement curve to initiate and propagate a peel fracture should be recorded. At least 30 mm of peel fracture should be established. The average peel force should then be determined unless there is a combination of adhesive and cohesive fracture or "stick-slip" (as illustrated in Figure 6). For these occasions, the mean lowest and the mean highest steady propagation peel force values should then be used to determine the adhesive and the cohesive fracture toughness values, respectively.

In order to conduct the corrections summarised in equations 6-10, it is necessary to measure tensile stress - strain plots on the material of the peel arms. (Two tensile stress-strain plots will be required if the materials of the peel arms are different). Establishing digitised, bilinear and elastic-power law plastic descriptions of the tensile stress-strain behaviour of the peel arms should be done as previously described in the section on fixed arm tests.
Calculation of the various adhesive fracture toughness terms is then done for each peel arm as described in the section on the fixed arm peel.

3.3 TEST REPORT FOR THE T-PEEL TESTS

The information required in a test report is similar to that for the fixed arm test. However, for the T-peel test there will be two peel arms to report and two possible specimen configurations (A and B) as well as the measured peel angle. However, there is no requirement to test both types of configuration. The information required is as follows:

General information
Name of Laboratory
Description of laminate
Material of peel arm
Adhesive used for gluing one arm to the peel table
Test equipment

Data from tensile test(s) on the peel arm material(s)
This should be provided for both peel arms (1 and 2) if different.
Test speed (mm/min)

Bilinear fit	**Power law fit**
Low strain modulus (E_1) (GPa)	Low strain modulus (E_1) (GPa)
Yield strain (%)	N
High strain modulus (E_2) (GPa)	Yield strain (%)
Alpha (E_2/E_1)	Yield stress (MPa)
Yield stress (MPa)	

Data from the peel test

Specimen configuration (A or B)
Measured peel angle (^{o}) [The smaller angle should be quoted]
Test speed (mm/min)
Specimen dimensions L (mm)
b (mm)
Peel arm thickness h (µm) (substrate only)
Thickness of adhesive layer h_A (µm)
(if $h_A > 0$ then conduct calculations for two cases $h_A = 0$ and the actual value for h_A)
Value ascribed to the modulus of the adhesive E_A (GPa)
Peel strength (P/b) (N/mm)

Derived results by calculations (for each of the three fits to the stress-strain data)
To be quoted for each peel arm
G (J/m^2)
G_P (J/m^2)
G_A (J/m^2)
Correction factor (%)

Overall results

$G_A = (G_A)_1 + (G_A)_2$

CONCLUDING COMMENTS

It would be helpful to use both test methods in order to determine the adhesive fracture toughness. Provided that the correction factor is not too large, then it can be hoped that the values will be the same for both test methods.
It is recommended that the software source used for the calculations is quoted in all reports.

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