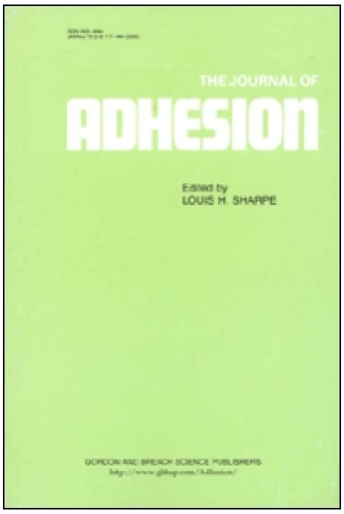

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Protocols for the Measurement of Adhesive Fracture Toughness by Peel Tests

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Protocols for the Measurement of Adhesive Fracture Toughness by Peel Tests

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This article reports on the work of the European Structural Integrity Society Technical Committee 4 (ESIS TC4) and its activities in the development of test protocols for peel fracture. Thirteen laboratories have been working on peel test methods in ESIS TC4 since 1997 and their activities are ongoing.

The aim of the work is to develop robust and credible test methods for the determination of adhesive fracture toughness by peel tests. Several geometric configurations have been used, namely, multi-angle fixed arm peel, T-peel, and roller assisted peel in the form of a mandrel test.

The starting point of their work is an established analysis of a peel method that is often developed from a global energy approach. The adopted analysis is combined with an experimental approach in order to resolve ambiguities in the determination of adhesive fracture toughness (G_A). The test methods involve the measurement of peel strength in order to calculate the total input energy for peel (G) and the calculation of the plastic bending energy (G_P) during peel. The latter is often obtained from a measurement of the tensile behaviour of the peel arm. Adhesive fracture toughness is then $G - G_P$.

Four ESIS TC4 projects are described. The first relates to fixed arm peel whilst the second and third involve both fixed arm and T-peel. The fourth project combines mandrel peel and fixed arm peel. Each project uses different types of polymeric adhesives in the form of quite different laminate systems. The selection of the laminate system enables all characteristics of laminate property to be embraced, for example, thin and thick adhesive layers, polymeric, and metallic peel arms and a range of flexibility in the laminates.

The development of the enabling science required to establish the test protocols is described and software for conducting all calculations is referenced.

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INTRODUCTION

Peel tests are used for the determination of adhesive strength of flexible laminate systems in aerospace, automotive, electronic, and packaging applications. Standard tests are available where peel strength (peel force per unit width of specimen) is measured. However, it is now well recognized [*e.g.*, 1,2] that the measurement of peel strength relates to a total input energy (G) for the peeling process and that an adhesive strength in the form of adhesive fracture toughness (G_A) can only be objectively determined when correction is made for the plastic bending energy (G_P) associated with the peel arm. A global energy analysis then gives:

$$G_A = G - G_P \tag{1}$$

Consequently, for most peel tests an experimental procedure will measure peel strength in order to calculate G and the tensile stress-strain properties of the peel arm material in order to calculate G_P. Equation (1) is used for the determination of adhesive fracture toughness but the analytical methods for determining G_P are often complex; although, software is available [3] and is based on the latest versions of the analysis [4]. In addition, other types of peel test (*e.g.*, a mandrel peel method) have been developed that enable G_A to be determined directly by experiment, although still based on a global energy analysis [5].

It would seem that the issues attendant on the determination of G_A are, therefore resolved. However, there can be a gap between established principles of measurement and a sound practical procedure.

One of the technical committees (TC4) of the European Structural Integrity Society (ESIS) has investigated this gap in relation to a number of test methods based on fracture mechanics principles. The aim of ESIS TC4 has been to establish test protocols by using groups of scientists from academy and industry in order to agree on a test method. It has tackled 17 different fracture tests on polymers, adhesives, and composites [6], and peel tests have been included in their activities.

The aim of this article is to describe the work of ESIS TC4 in relation to peel tests. In particular the development of peel test protocols for the determination of G_A *via* fixed arm peel, T-peel, and mandrel peel. This will be achieved by first outlining the principles of analysis for these three methods and then describing the issues that needed attention in order to establish the test protocols. ESIS TC4 conducts its activities through round robin experimental projects and the outcomes of four individual round robins will be included in this article. The types of laminate used in the studies also included polymer-polymer films, polymer-thin metal laminates, polymer-metal laminates with thick polymer adhesives, as well as thick metal substrates.

ANALYSIS OF PEEL TESTS

Fixed Arm and T-Peel Tests

Equation (1) is used to calculate adhesive fracture toughness for both fixed arm and T-peel methods. Full details of analysis are given elsewhere [1,4] but the salient steps are now summarized. Figure 1 shows the peel parameters as illustrated for the fixed arm method.

P is the peel force, θ is the peel angle, h is the peel arm thickness (without adhesive coating), h_a is the adhesive bond-line thickness, R_0 is the radius of curvature at the root, and θ_0 is the root rotation angle. Total external energy is given by (neglecting any tensile deformation of the peel arm):

$$G = \frac{P}{b}(1 - \cos\theta) \tag{2}$$

where b is the width of the peel arm.

The plastic bending energy of the peel arm is given by:

$$G_P = \frac{E\varepsilon_y^2 h}{2} f(k_0) \tag{3}$$

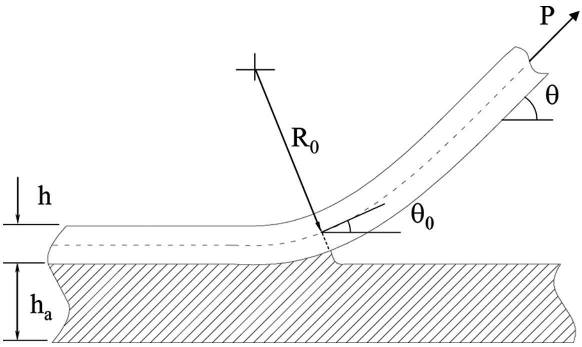


FIGURE 1 Peel parameters in a fixed arm test.

where E is the tensile elastic modulus of the peel arm, ε_y is the yield strain of the peel arm, and $f(k_0)$ is a function based on a normalized curvature, k_0, *i.e.*;

$$k_0 = \frac{h}{2\varepsilon_y}\frac{1}{R_0} \tag{4}$$

The function $f(k_0)$ also depends on the work hardening coefficient derived from measurement of the tensile stress-strain properties of the peel arm material. These coefficients (α and N, respectively) depend on whether a bilinear fit or a linear power law fit is used to describe the experimental stress-strain data. Up to the yield point

$$\sigma = E\varepsilon \tag{5}$$

At deformations beyond yield, the bilinear fit is described by

$$\sigma = \sigma_y + \alpha E(\varepsilon - \varepsilon_y) \tag{6}$$

And for the linear power law fit

$$\sigma = \sigma_y\left(\frac{\varepsilon}{\varepsilon_y}\right)^N \tag{7}$$

where σ_y is the yield stress and σ and ε are stress and strain, respectively.

This analytical approach is used to determine the adhesive fracture toughness for fixed arm peel [6], where a software procedure known as *ICPeel* [3] aids the calculations. A similar analysis is also used for T-peel, but because there are now two peel arms, the calculations are conducted twice and the sum of the individual values provide a final value for G_A [6]. For T-peel tests it is recognized that one peel arm will be stiffer than the other and, hence, peel angles are seldom 90° [6–8]. When the stiffer arm is at the bottom of the test configuration, the unpeeled section will point upwards; this is designated configuration A. When the stiffer segment is at the top, the unpeeled section points downwards and the configuration is designated B. Both versions are used in the experimental work and are shown in Appendix 2.

Mandrel Peel Tests

The configuration of a mandrel peel procedure is shown in Figure 2. D is an alignment load, θ_1 is the angle between the vertical and the peel force (P) and R_1 is the radius of the mandrel. In our experimental arrangement θ_1 is 90°.

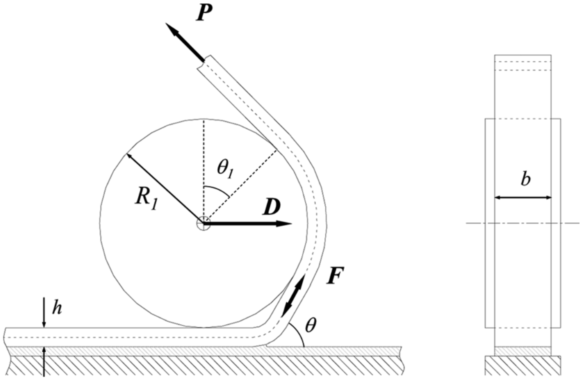


FIGURE 2 Configuration for a mandrel peel test.

If frictional effects are negligible (although in practice this is a reasonable assumption, in reality the friction coefficient μ can be calibrated out), then a global energy analysis provides an expression for G [5]:

$$G = G_A + G_P = \frac{P}{b} - \frac{D}{b} \tag{8}$$

Therefore, G_A can be directly determined by experiment. Two experiments are conducted, first with an unbonded laminate ($G_A = 0$) and second with a bonded laminate. Figure 3 shows how the data are presented and how G_A is determined.

In general, results are presented as plots of G_A *versus* alignment force per unit width (D/b). This approach accommodates results from

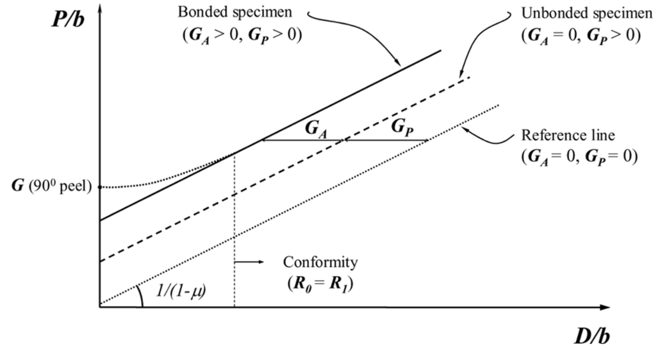


FIGURE 3 Presentation of results for mandrel peel.

a mandrel procedure as well as results by other approaches (fixed arm peel and cohesive fracture tests) and enables all results to be presented together.

EXPERIMENTAL RESULTS

Preamble

Peel projects have been active in ESIS TC4 since 1997 and are still ongoing. Table 1 summarizes the various activities.

In all 13 laboratories have participated in the work and are listed in Appendix 1. The first project was aimed at conducting fixed arm peel tests on a polyethylene aluminium coated packaging laminate (PE/AA) where the peel arm was polyethylene. Eight laboratories were involved. The second project was based on a polypropylene/ethylene vinyl alcohol/polypropylene laminate (PP/EVOH/PP) where tests involved both fixed arm and T-peel. Seven laboratories participated. Detailed results from these first two round robin projects have been published [7,8], therefore, only outline comments need to be included now.

The third project has involved an aluminium alloy/polypropylene/aluminium alloy laminate (AA/PP) and, although only four laboratories participated, it was possible to address the issues that arose in the first two round robins together with a number of additional aspects. The results on AA/PP will be presented in detail and relate to both fixed arm and T-peel methods.

The fourth project started in 2004 and is as yet incomplete. It is based on an aerospace system with an aluminium alloy/epoxy/aluminium alloy laminate (AA/FM73) and involved a new peel method based on mandrel peel together with fixed arm peel. Early results from this project will be presented and eight laboratories are participating.

In all of the projects the aim has been to agree on a test protocol. Consequently, the presentation of results is conducted with an eye on illustrating the ambiguities and issues that are commonly experienced

TABLE 1 Summary of ESIS TC4 Peel Projects

Round robin project	Time period	No. of laboratories	Laminate	Test and specimens
1	1997–1999	8	PE/AA	Fixed arm
2	2000–2002	7	PP/EVOH/PP	Fixed arm and T-peel
3	2003–2006	4	AA/PP	Fixed arm and T-peel
4	2004–present	8	AA/Epoxy	Fixed arm and mandrel peel

by laboratories when attempting to determine adhesive fracture toughness. As was pointed out earlier the strategic and analytical aspects were agreed on from the outset, but the practical interpretations and problems only become apparent by comparative experimental studies.

Round Robin 1 Based on PE/AA Laminates

The first project involved eight laboratories conducting fixed arm peel tests on an aluminium-polyethylene (AA/PE) packaging laminate and only applying a bilinear fit to the tensile stress-strain results. Results in terms of total external energy to peel (*G*) and adhesive fracture toughness are shown are shown in Figure 4 from 90° fixed arm peel tests.

Good agreement was achieved for *G*, but there was greater scatter for G_A. The tensile stress-strain curve for the polyethylene peel arm was clearly bilinear in character [7], but the position of the yield coordinates did allow different interpretations, mainly due to the extent of deformation recorded before the test was stopped. Consequently, there was considerable scatter in the values of yield strain [7] and this accounted for the scatter in G_A.

Round Robin 2 Based on PP/EVOH/PP Laminates

In the second round robin, on PP/EVOH/PP laminate, both fixed arm and T-peel experiments were conducted and again only a bilinear fit

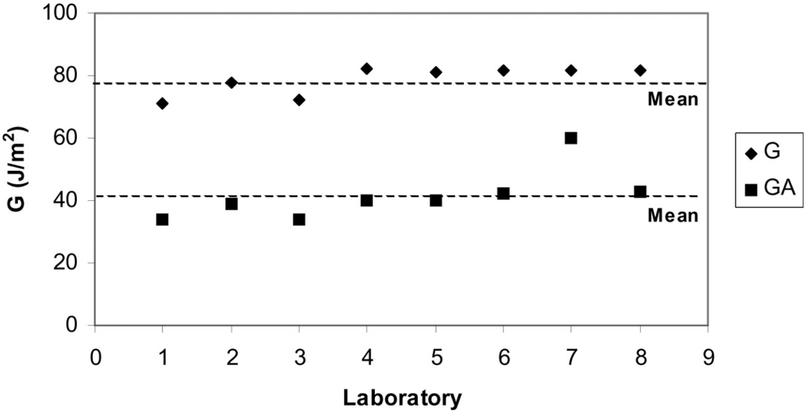


FIGURE 4 Total peel energy (*G*) and adhesive fracture toughness (G_A) for AA/PE laminate with a 200 μm PE peel arm from 90° fixed arm peel tests.

TABLE 2 Summary of Results from Round Robin 2 on PP/EVOH/PP Laminates

Test method	G_A (J/m^2)
Fixed arm peel at 90°	215 (47)
Fixed arm peel at multiple angles between 45° and 135°	206 (42)
T-peel	211 (56)

was used to describe the tensile stress-strain behaviour of the 75 μm EVOH/PP peel arm. Values of G_A gave encouraging agreement; Table 2 summarizes the data with standard deviations in parenthesis. The standard deviations are large and indicate scatter in the data. This was again due to variations in the fitting of a bi-linear function to the stress-strain data for the peel arm material and, in particular, to variations in yield strain and the work hardening coefficient, α [8]. Investigation showed that each laboratory used a slightly different approach in the determination of yield coordinates. For example, it was found that the linear portions of the stress-strain curves were fitted before definition of the yield coordinates. This is contrary to the demands of Equations (5) and (6) where it is implicit that the procedure should define the elastic modulus from Equation (5), then determine the yield coordinates and then fit Equation (6).

The first two peel round robin projects suggested that obtaining peel fracture data could be achieved with high consistency, but that determination of the stress-strain behavior of the peel arm material and its subsequent use in the calculation of plastic bending energy was more problematic.

Round Robin 3 on AA/PP Laminate

Aims

The choice of laminate for the third round robin project was quite different. Two aluminium alloy sheets (AA 5154–O) with a thickness of 0.24 mm were bonded with a polypropylene adhesive with a notational thickness of 0.95 mm. Therefore, the overall thickness of laminate was 1.43 mm so that the bond-line thickness was substantial and allowance would need to be made for deformations of the adhesive. (A modulus was assigned for the adhesive, $E_a = 1.5$ GPa, as this was typical for this type of polypropylene, and the deformation of the adhesive layer as described in Reference 4 is incorporated into the *ICPeel* software for the calculations).

The third round robin aimed to determine adhesive fracture toughness by both fixed arm peel and T-peel and special attention was given to three areas of testing and analysis:

(1) Determination of stress-strain behaviour of the AA peel arm where a bilinear and linear power law fit were used.
(2) The calculation of G_P and the value to use for the peel arm thickness.
(3) The influence of bond-line thickness on the calculation of G_A.

A set of experimental results from each laboratory is given in Appendix 2 and results for adhesive fracture toughness are presented in the following sections.

Parameters from Tensile Testing of the Peel Arm

The determination of the tensile stress-strain behavior of the peel arm material followed the experimental requirements of previous protocols [6]. The tensile test had to 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 10-mm width and 100-mm length. In addition, an extensometer was required to measure the strain deformations necessary to define the elastic deformations. The extensometer, ideally, should be of a noncontacting type because 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, if possible.

The fitting of the bilinear and the linear power law functions was defined in more detail. First, it was necessary to define the elastic modulus of the peel arm (E). Second, the coordinates of the yield point were defined by conducting a linear fit to early plastic data to meet the elastic modulus line. This defined the yield coordinates (σ_y, ε_y) and these coordinates were used for fitting the plastic curve whether linear or power law. The stress-strain parameters (E, σ_y, α and N) could then be obtained. Figure 5 shows an alleged correct fitting of bilinear and linear-power law fits to the experimental stress-strain data for the peel arm material. (The power law fit uses Equation [7], which is a two-parameter model. It is possible that a three-parameter model would improve the fit since it would not force the data through the origin. However, for the time being, it is Equation [7] that is used although other options might be possible in the future.)

Three laboratories reported data according to this procedure and these are shown in Table 3. (The fourth laboratory did not have access to an appropriate extensometer for measuring strain in the elastic

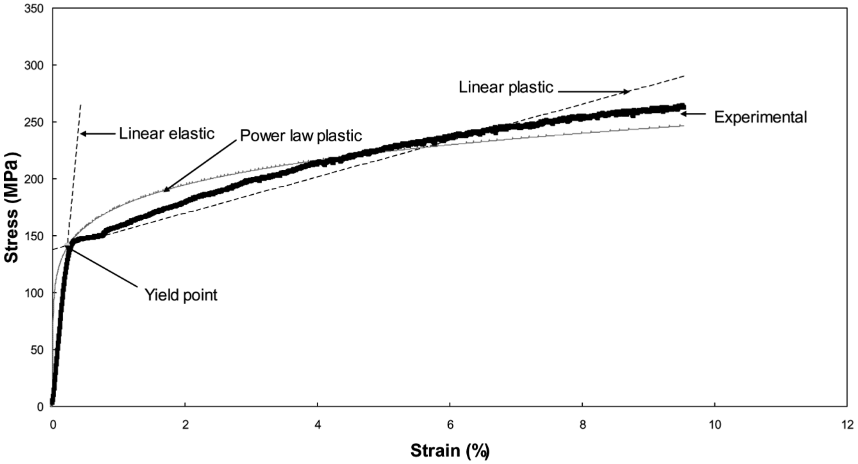


FIGURE 5 Experimental stress-strain data for AA 5154-O with fits for bilinear and linear power law functions correctly applied.

part of the curve and, consequently, used the data from another laboratory in order to complete their calculations of G_A).

Although only three laboratories measured the stress-strain parameters, the results in Table 3 give an indication of the likely experimental scatter associated with systematic errors in the measurements. These ranges can be used in order to calculate the consequential variations in G_A; this has been done for three parameters, namely E, σ_y and α (ε_y and N are ignored because they are essentially repeats of σ_y and α, respectively.) Experimental scatter for these parameters (and designated "interlabs" range) is taken as:

E (60–75) GPa
σ_y (140–152) MPa
α (0.020–0.032)

TABLE 3 Results for Fitting Bilinear and Linear Power Law Functions to Tensile Stress-Strain Data for Peel Arm AA 5154–O

Laboratory	E (GPa)	σ_y (MPa)	ε_y (%)	α	N
1	70	140	0.21	0.027	0.16
2	62	141	0.23	0.026	0.15
4	67	151	0.22	0.030	0.12

where α is the work hardening factor from a bilinear fit and equals E_P/E, where E_P is the plastic modulus and N is the power law integer from the power law fit.

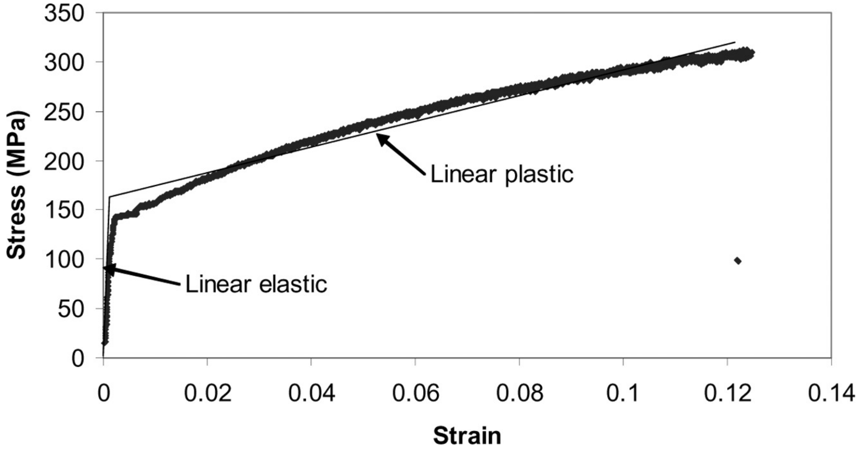


FIGURE 6 A possible incorrect procedure for fitting a bilinear function to stress-strain data for AA 5154–O peel arm.

Of course, if the procedural requirements of the test protocol are not met, then other ways of fitting the bilinear function or the linear power law function would also be possible. These incorrect approaches are illustrated by example in Figures 6 and 7. In both cases the coordinates of the yield point are not defined before fitting the curves. Such approaches are possible because the curve fitting appears more satisfactory but the yield coordinates are incorrect.

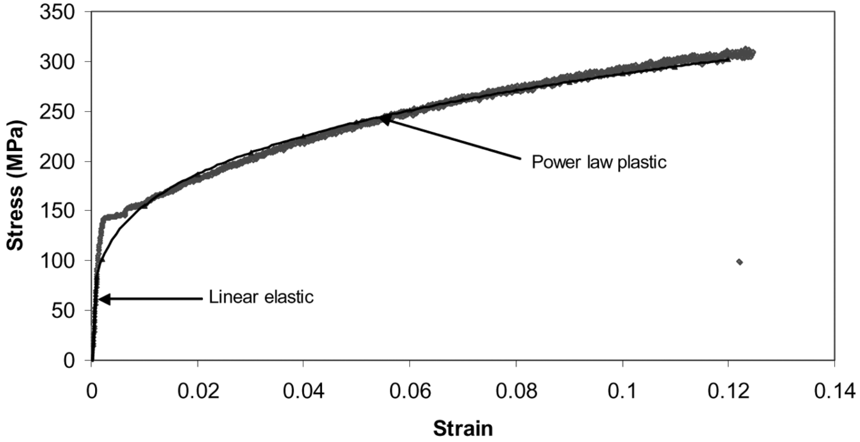


FIGURE 7 A possible incorrect procedure for fitting a linear power law function to stress-strain data for AA 5154–O peel arm.

Incorrect possibilities for fitting the stress-strain curves lead to a different and wider range of values for the three parameters E, σ_y and α. A possible range is given below:

E (40–90) GPa
σ_y (100–190) MPa
α (0.00–0.060)

These values of tensile stress-strain parameters can also be used for calculating the values of G_A and these data are designated "wide range." When conducting such calculations a set of data from laboratory 1 was used.

Figures 8 to 10 show the influence that the "interlabs" and "wide range" versions of these parameters have on calculated G_A, where the Y-axis scale is the same for each plot.

The influence of elastic modulus is the least significant, whilst the work hardening coefficient (from a bilinear fit) seems the most significant. However, it is possible that the range selected for α is less realistic than that for yield stress. For the "interlabs" range the scatter of G_A about the mean values is quite small, being 0.4% for modulus, 1.3% for yield stress, and 3.4% for work hardening coefficient. If these were possible experimental errors for the determination of G_A then they would be considered negligibly small.

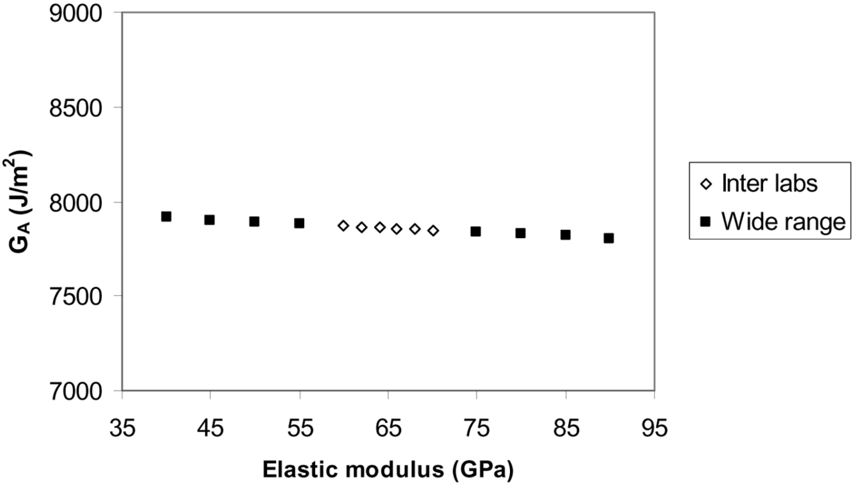


FIGURE 8 The influence of elastic modulus (E) on the calculated value of G_A (values of $\sigma_y = 140$ MPa and $\alpha = 0.027$ are used).

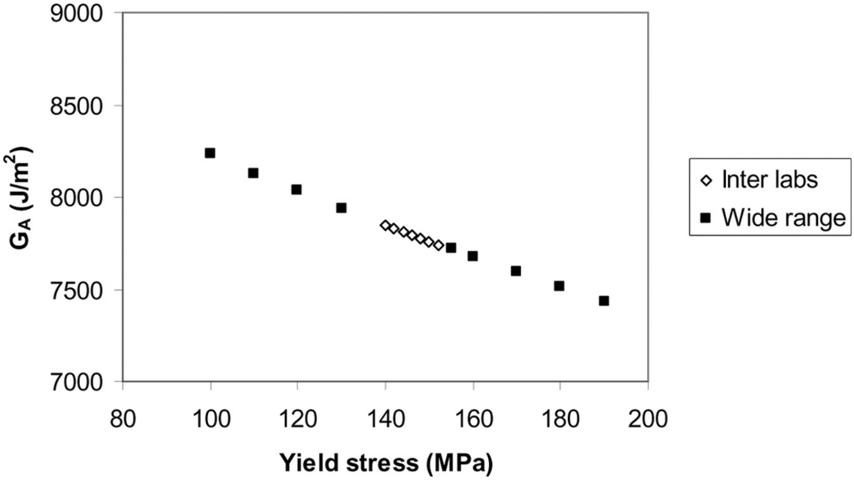


FIGURE 9 The influence of yield stress (σ_y) on the calculated value of G_A (values of $E = 70$ GPa and $\alpha = 0.027$ are used).

The influence of the "wide range" parameters on G_A is relatively large at about 20%. Therefore, it is important to ensure that the correct procedure is used in the determination of the stress-strain parameters in order to obtain values in the "interlabs" range.

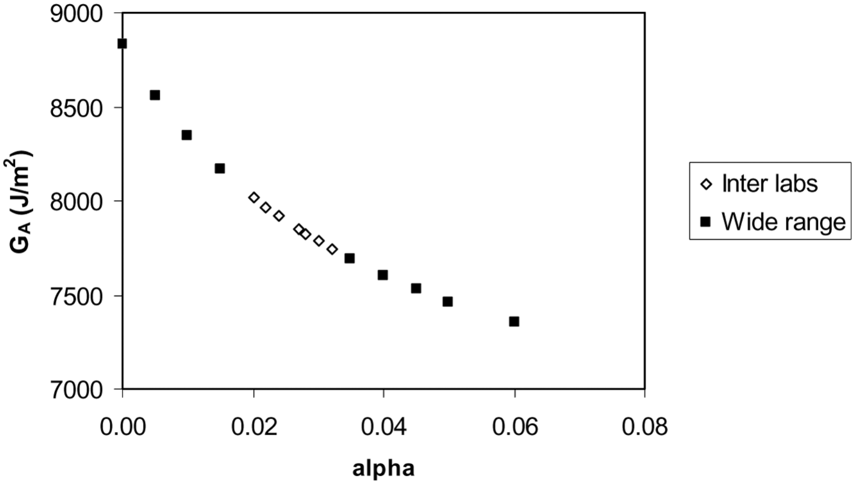


FIGURE 10 The influence of work hardening coefficient (α from bilinear analysis) on the calculated value of G_A (values of E = 70 GPa and $\sigma_y = 140$ MPa are used).

Definition of Peel Arm Thickness

The peel arm thickness (h) is used in the calculation of modulus from tensile stress-strain results and also in the determination of G_A from the peel test. In both cases the value of h should relate to the thickness of the metal peel arm and should not include any aspect of an adhesive coating on the peel arm. Therefore, if the peel arm thickness is measured after the peel test when there may exist an adhesive coating on the peel arm, then an error may be introduced.

ICPeel may be used in order to investigate the influence of apparent peel arm thickness on the value of G_A and results of calculations for the AA/PP laminate are shown in Figure 11. If it is assumed that the correct value of h is 0.24 mm, but with an adhesive of thickness 0.95 mm, then it is conceivable that an apparent value of h could be quite large if a thick layer of adhesive remains on the peel arm. For example, laboratories reported adhesive coating thickness of 0.1 mm. Therefore, if h had been assigned a value of 0.34 mm, then G_A would be 13% too small.

The Influence of Adhesive Thickness

The choice of laminate in the third round robin project provided a system with a significantly thick adhesive. In the first two projects the adhesive thickness was negligibly small. However, with the total laminate thickness of 1.43 mm and a notional adhesive thickness of

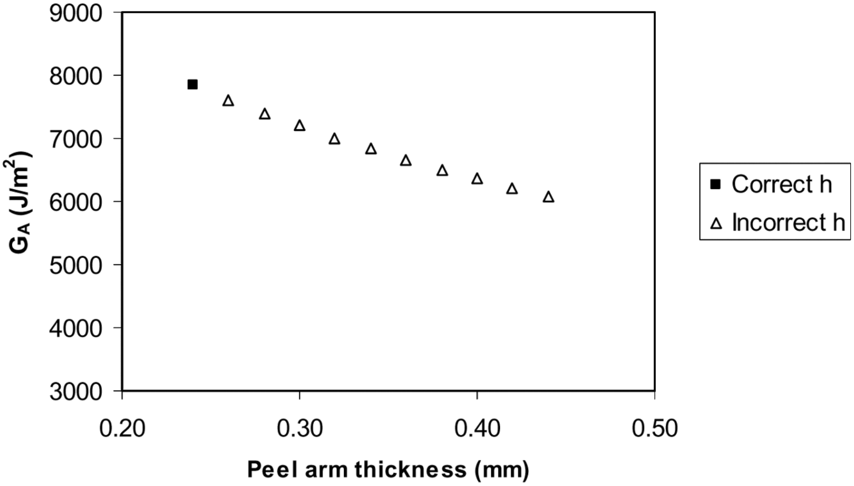


FIGURE 11 Influence of peel arm thickness on G_A for AA/PP laminates. Calculations are based on parameters $E = 70$ GPa, $\sigma_y = 140$ MPa, and $\alpha = 0.027$.

0.95 mm the deformation of the adhesive was likely to be an important factor in the peel process [4]. A relative thickness of adhesive of this magnitude is an extreme case and will fully challenge the calculations in *ICPeel*.

The work of Reference [4] used either a simple linear-elastic stiffness approach or a critical limiting maximum stress approach in order to provide an analytical elastic-plastic model of the peel test. Both approaches gave values of adhesive fracture toughness which are independent of the details of the test geometry. Using the linear-elastic approach, they demonstrated that a finite thickness of adhesive level could also be incorporated into the model where it was necessary to have knowledge of two additional parameters, the thickness and modulus of the adhesive (h_a and E_A, respectively).

ICPeel software [3] is based on the analysis of Reference [4] and has the ability for a user to select the value of adhesive thickness (h_a) for the calculations. Consequently, each participant in this project conducted calculations based on two values of adhesive thickness, namely $h_a = 0$ and $h_a = 0.95$ mm (or whatever value they measured for h_a as given in Appendix 2). A value of $E_A = 1.5$ GPa was used, as mentioned earlier.

Figure 12 shows the results for fixed arm peel at 90°. There is a large difference between the values with the mean $G_A = 8229$ (704) J/m^2 when adhesive thickness is included, but with a mean value of $G_A = 4211$ (359) J/m^2 when adhesive thickness is taken as

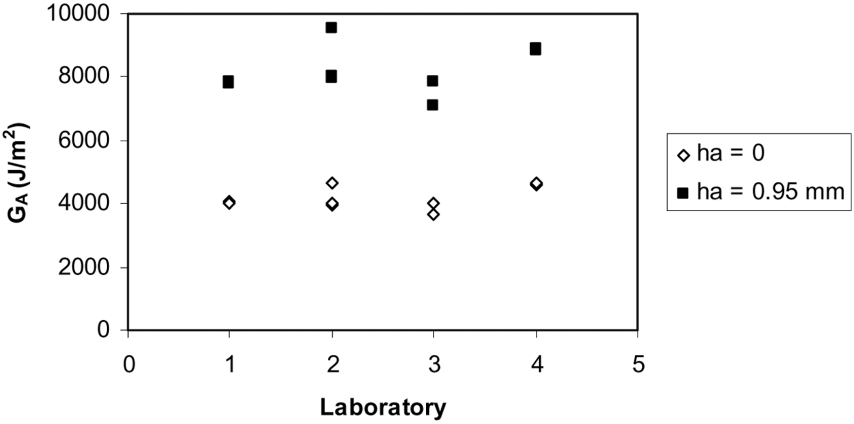


FIGURE 12 Values of G_A for AA/PP laminate by 90° fixed arm peel with two different values of adhesive thickness (h_a). Analysis is based on a bilinear fit to the stress-strain results.

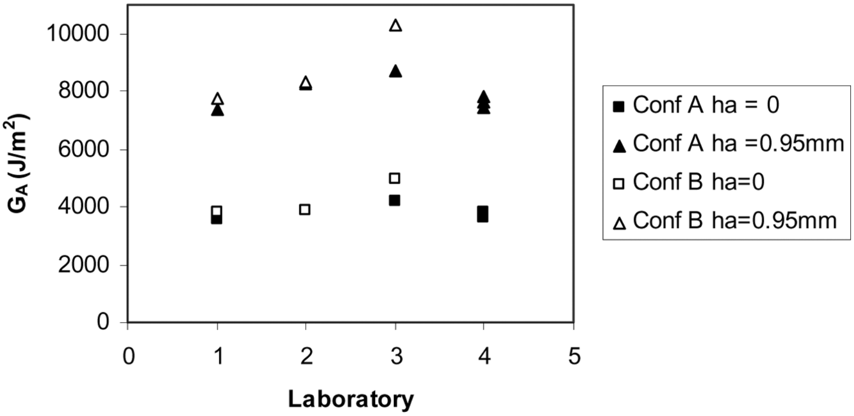


FIGURE 13 Values of G_A for AA/PP laminate by T-peel with two different values of adhesive thickness (h_a). Analysis is based on a bilinear fit to the stress-strain data. Configuration A has the unpeeled specimen pointing upward because the stiffer segment is at the bottom and configuration is *vice versa* [6,8].

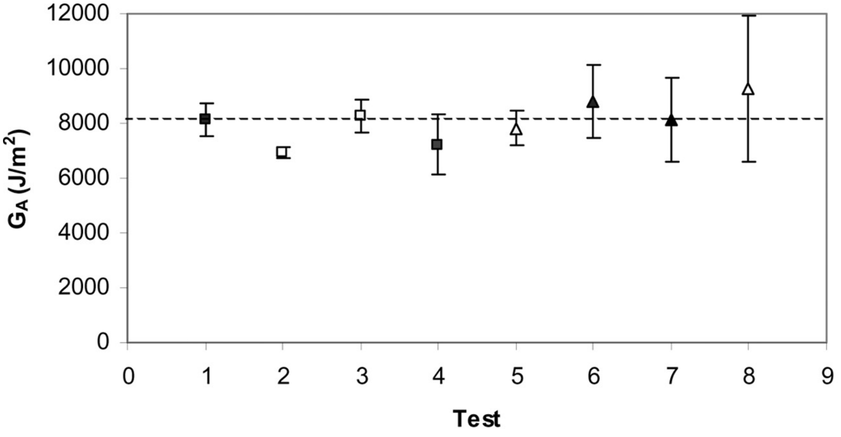
zero (standard deviations in parenthesis). There will be significant deformations in the adhesive layer and, therefore, correct values of G_A are taken as those where the notational $h_a = 0.95\,\text{mm}$.

Figure 13 shows the T-peel results for the two sets of calculations, *i.e.*, with $h_a = 0$ and 0.95 mm. A similar story emerges, although one of the sets of results from laboratory 3 appears to be an outlier. If this result is neglected, then, when adhesive thickness is included, a mean of $G_A = 8000\ (370)\,\text{J/m}^2$ is obtained, but with a mean value of $G_A = 3830\ (160)\,\text{J/m}^2$ when adhesive thickness is taken as zero (standard deviations in parenthesis). These results agree well with those results obtained by fixed arm peel. Again, it shows the importance in accommodating deformations in the adhesive layer.

Overview of Results from Round Robin 3 for AA/PP Laminate

All results by bilinear and linear power law modelling of fixed arm and T-peel data are shown in Figure 14. They are in the form of G_A values plotted against test method with a key provided.

Error bars are ± standard deviations and it is noticeable that the scatter is not consistent with most scatter associated with some of the T-peel tests. However, it was noted that laboratory 3 seems to have some outliers. Agreement for the G_A values for each method is remarkably good and gives high confidence to the refinements that have been made to the protocol as discussed in the last three sections.



1	2	3	4	5	6	7	8
Fixed 90° Bilinear	Fixed 135° Bilinear	Fixed 90° Power law	Fixed 135° Power law	T Peel A Bilinear	T Peel B Bilinear	T Peel A Power law	T Peel B Power law

FIGURE 14 Overview of results by all combination of methods. Mean value of G_A plotted as dashed line and error bars are ± standard deviations.

An adequate test protocol has, therefore, been established for the measurment of G_A by the two test geometries.

Round Robin 4 Using Mandrel and Fixed Arm Peel with AA/FM73

Aims

A roller assisted peel test such as mandrel peel [5] provides direct experimental determination of G_A and eliminates any complexities associated with elaborate analytical procedures (such as those embedded in fixed arm and T-peel) and in addition does not require knowledge of the stress-strain parameters from tensile deformation of the peel arm. Therefore, the aim of this stage of the work was to measure G_A by mandrel peel and to compare results from the developed procedure for fixed arm peel. This is eased experimentally by using a mandrel peel instrument (for example as described in Reference 5) where both mandrel and 90° fixed arm peel can be conducted on the same specimen and on the same instrument.

Eight laboratories are participating in a round robin on mandrel peel using an aerospace type laminate (0.635 mm AA 2024–T3/FM73). Each laboratory is using the same mandrel peel instrument. The aim is to measure G_A by mandrel peel and also by 90° fixed arm peel. In addition, G_C (cohesive fracture toughness) is measured using a tapered double cantilever beam specimen (TDCB) [9]. Results from only one laboratory are currently available and are briefly reported.

Mandrel and Fixed Arm Peel Results

In order to conduct a mandrel peel test according to the analysis presented earlier it is vital to ensure that the peel arm conforms to the mandrel roller during the peel process and this condition is satisfied when $R_0 \geq \mathrm{R}_1$. R_0 is the radius of curvature of the peel arm and R_1 is the radius of the mandrel roller.

Figure 15 illustrates the type of instrument that can be used for mandrel peel tests. Full details are given elsewhere [5,10]. The figure shows the configuration for a mandrel test where the mandrel roller can be selected from a range with radii of 1 mm to 20 mm. In this version of the instrument the mandrel roller and holder can be removed and this converts the set-up to a 90° fixed arm peel test without change to the specimen design. An ESIS TC4 protocol has been written for procedures with the instrument operating as a mandrel and fixed arm device and based on a pending publication [11].

Therefore, the previous protocol for fixed arm peel is used for obtaining G_A and *ICPeel* is used to calculate R_0. It is then possible to select a mandrel roller radius to satisfy the condition that $R_0 \geq R_1$ and, hence, achieve conformance of the peel arm to the mandrel roller. A 5-mm mandrel roller was selected for the work of all laboratories.

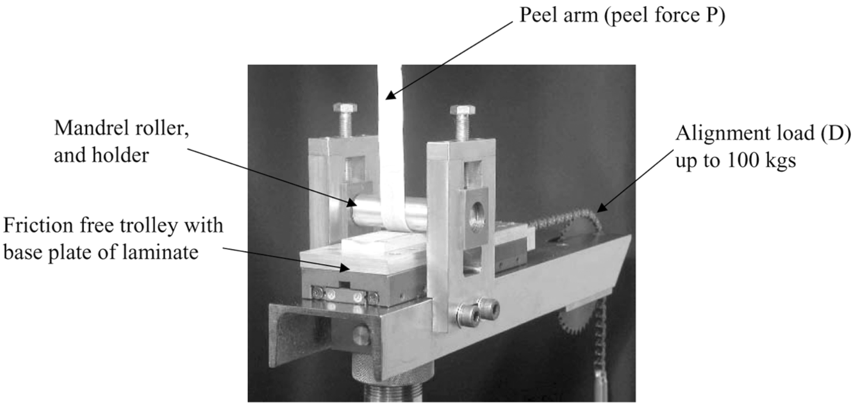


FIGURE 15 A mandrel peel instrument showing the peel force and alignment force.

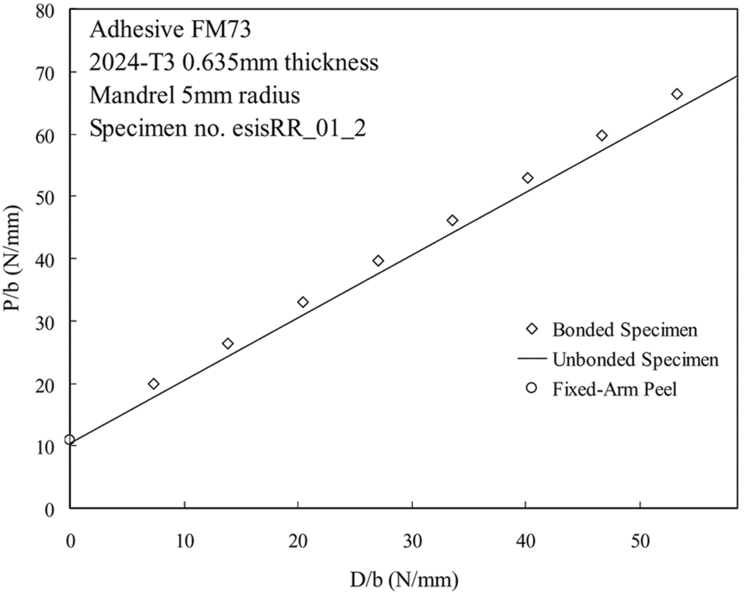


FIGURE 16 Mandrel peel results for bonded and unbound specimens as well as fixed arm peel.

Figure 16 shows the mandrel results for bonded and unbonded specimens and the analysis section presented earlier describes the determination of G_A and G_P. The fixed arm peel data are also included. The fixed arm data are analysed with *ICPeel* software [3] that also provides a calculation of the radius of curvature (R_0) for the peel arm and a value of 6.9 mm is obtained. Consequently, the selected value of mandrel roller radius ($R_1 = 5\,\text{mm}$) satisfies the condition that $R_0 \geq R_1$ and, therefore, conformance of the peel arm can be expected during peel in the mandrel test.

It is generally more helpful to present results as G_A *versus* D/b and this is done for the data in Figure 16 by the plots in Figure 17. The fixed arm data and the results from the TDCB tests are also shown in Figure 17. All peel fractures and other fractures are cohesive, *i.e.*, the crack is growing in the adhesive material.

With reference to Figure 17 it is clear that there is perfect agreement between G_C from TDCB and G_A from fixed arm peel. In addition, the mandrel data for G_A agree with both other sets of data. Consequently, for cohesive fracture there is good agreement between G_C and G_A. However, of greater relevance is that the established protocol for fixed arm peel is providing data that agree with the adhesive

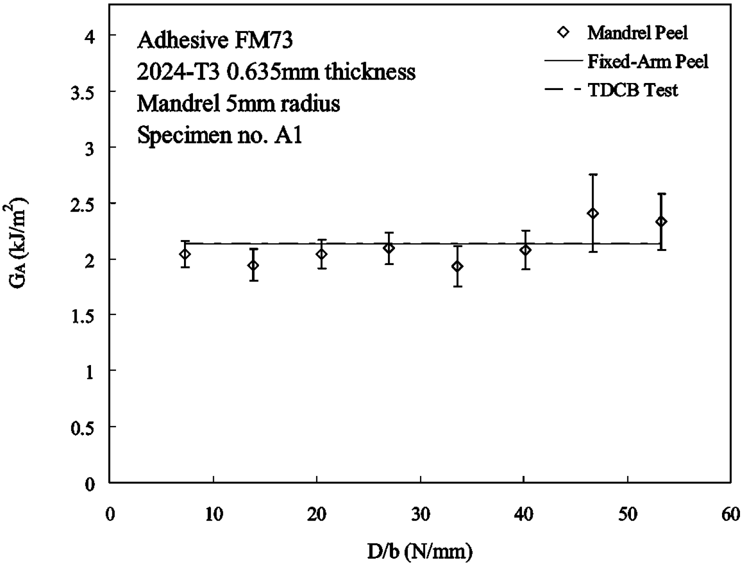


FIGURE 17 Results from mandrel peel with a roller radius of 5 mm and where R_0 is 6.9 mm. Results are plotted as G_A *versus* D/b and include data from fixed arm peel and TDCB.

fracture toughness results from the emerging mandrel protocol. We await the results for the other seven laboratories before making further comment.

CONCLUSIONS

A global energy analysis (reported in the literature) has provided a strategy for determining adhesive fracture toughness in peel and involves subtracting the plastic bending energy during peel from the total energy required to peel. Although the analysis is elegant and credible, it does not provide the practical means for determining G_A, since a number of experimental and interpretational factors can lead to problems. A group of 13 laboratories based throughout Europe have been collaborating on the development of test protocols to promote the analysis into test methods. Their work has involved four projects, three of which are complete and one remains active.

In the first project a PE/AA laminate was used to determine G_A by fixed arm peel. They reported good consistency in the measurement of peel strength (P/b) and calculation of total peel energy (G) indicating few problems in the conduct of the peel tests. However, there was significant scatter for the determination of plastic bending energy and, consequently, this scatter was also reflected in the determination of G_A.

In the second project a PP/EVOH/PP laminate was used in both fixed arm and T-peel tests. Again, there was good consistency in the peel tests for the measurement of peel strength, but scatter was introduced when determining G_A from the total input energy. Specific problems were identified in the measurement of the tensile behavior of the peel arm and in fitting a bilinear function to the data.

The third project addressed these issues with modifications to the protocol and in the third round robin quite a different laminate was used. An AA/PP laminate had a thick adhesive layer where deformations of the adhesive would need accommodating. In addition, the tensile behavior of the peel arm was fitted to both a bilinear function and a linear power law function. It was necessary to define the yield coordinates before fitting the model. Good agreement was achieved in the measurement of G_A from this work by all of the method combinations. Moreover, a detailed sensitivity analysis gave an indication of the scatter that can be expected from systematic errors and from possible operational errors.

The fourth project is conducted on AA/FM73 laminate with the aim of comparing G_A from a mandrel method with that from fixed arm peel. Early results are encouraging.

The work of ESIS TC4 in its quest to develop test protocols relating to fracture demonstrates that a robust test method is not established until both analysis and experimental procedures are developed and combined.

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REFERENCES

[1] Kinloch, A. J., Lau, C. C., and Williaims, J. G., *Int. J. Fracture* **66**, 45–70 (1994).

[2] Aravas, N., Kim, K. S., and Loukis, M. J., *Materials Sci. Eng.* **A107**, 159–168 (1989).

[3] Imperial College and http:/www.me.ic.ac.uk/AACgroup/ peel test protocols-ICPeel

[4] Georgiou, I., Hadavina, H., Ivankovic, A., Kinloch, A. J., Tropsa, V., and Williams, J. G., *The Journal of Adhesion* **79**, 239–265 (2003).

[5] Kawashita, L. F., Moore, D. R., and Williams, J. G., *The Journal of Adhesion* **80**, 147–167 (2004).

[6] Moore, D. R., Pavan, A., and Williams, J. G., editors, *Fracture Mechanics Testing Methods for Polymers, Adhesives and Composites* (Elsevier, Oxford, UK, 2001).

[7] Moore, D. R. and Williams, J. G., *The Measurement of Peel Strength and the Determination of the Interfacial Work of Fracture for Flexible Laminates* (231 Fracture of Polymers, Composites, and Adhesives, ESIS pub **27**, Elsevier, 2000).

[8] Moore, D. R. and Williams, J. G., *The Determination of Adhesive Fracture Toughness for Laminates by the Use of Different Test Geometry and Consideration of Plastic Energy Correction Factors* (Fracture of Polymers, Composites, and Adhesives, ESIS pub **32**, Elsevier, 2003).

[9] Kawashita, L. F., Moore, D. R., and Williams, J. G., *Composites Interfaces* **12**, 837–852 (2005).

[10] Kawashita, L. F., Moore, D. R., and Williams, J. G., *Journal of Adhesion* **81**, 561–586 (2005).

[11] Kawashita, L. F., Moore, D. R., and Williams, J. G., A Critical Investigation of the Use of a Mandrel Peel Method for the Determination of Adhesive Fracture Toughness to be published in *Engineering Fracture Mechanics* (2006).

APPENDIX 1: PARTICIPANTS IN THE ESIS TC4 PEEL ACTIVITIES

ICI plc, UK (D. R. Moore, R. S. Hardy)
Imperial College, London (L. F. Kawashita, D. R. Moore, J. G. Williams)
University College Dublin (N. Murphy)
Elf-Atochem, France (J. Pascal)
ATO-DLO, Holland (H. Bos)
University of Twente, Holland (P. E. Reed)
BASF, Germany (F. Ramsteiner, H. Steininger)
DuPont S.A., Switzerland (S. Ducret)
Martin-Luther University, Germany (H. W. Grellman)
Politecnico de Milano, Italy (A. Pavan, S. Fara, R. Rizzieri)
University of Kaiserslautern, Germany (J. Karger-Kocsis)
Tetra-Pak, Switzerland (P. Emery)
Insa de Lyon, France (A. A. Roche, deceased)

APPENDIX 2: EXPERIMENTAL DATA FROM EACH LABORATORY FOR ROUND ROBIN 3

Table 4 provides one set of experimental results from round robin 3 for each laboratory for both fixed arm and T-peel. This is given in order to indicate typical measurements that each laboratory made in their peel tests. Table 3 in the main body of the text shows results for the tensile stress-strain measurements on the peel arm material.

Of course, each laboratory made more measurements that those given in Table 4; however, all results in the form of derived adhesive fracture toughness are given in the main body of the text.

TABLE 4 One Set of Experimental Peel Results from Each Laboratory from Round Robin 3

	General dimensions			Fixed arm peel		T-peel		
Laboratory	h_a (μm)	h (μm)	b (mm)	P (N)	θ (°)	Config.	P (N)	θ (°)
1	890	240	20.0	251	90	A	155	36.5
2	950	240	20.0	252	90	A	165	37.2
3	950	240	20.0	250	90	A	174	39.0
4	930	240	20.0	279	90	A	163	35.0

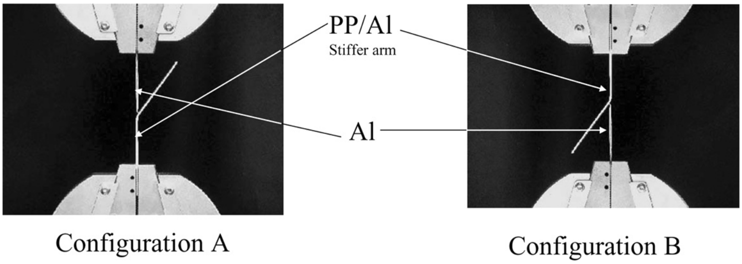

FIGURE 18 Two test configurations for T-peel experiments.

In the T-Peel tests it has already been mentioned that there are two test configurations (A and B). Figure 18 shows photographs of both configurations for experiments conducted with the AA/PP laminates.