

Coupon Tests on z-Pinned and Unpinned Composite Samples for Damage Resistant Applications

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Summary: The main objective of the work was to determine the mechanical properties of Z-pinned and unpinned composites samples. Tensile and interlaminar fracture tests under mode I have been carried out on thermoset polymer matrix composite samples composed of CYTEC 977-2 and carbon fibre yarn HTA-12K/35. The presence of the Z-pins has been found to significantly increase the interlaminar fracture toughness. The crack resistance improvement has been found to be accompanied by a small reduction of the in-plane stiffness properties for a Z-pins density of 2%. The results are presented in relation to the size and pinning quality and are rationalised on the basis of the location of Z-pin block within the sample.

Keywords: composites; interlaminar fracture toughness; Z-pin

Introduction

The primary weakness of laminated composites is their low stiffness and strength in the through-thickness direction, making interlaminar delaminations a major concern in the application of polymer matrix continuous-fiber-reinforced composites. In recent years a new technology, consisting of through-the-thickness reinforcement by Z-Fiber pinning, has been attracting considerable attention and industrial interest.^[1] This technique is a newly available alternative to the stitching of laminates in the z-direction, which had in the past produced some promising improvement in the damage tolerance of polymer matrix composites.^[2]

The Z-fibers, hereafter referred to as Z-pins, are inserted orthogonally to the plane of the composites plies during the manufacturing process, before the resin

matrix is cured, effectively pinning the individual layers together.

Figure 1 shows the reinforcing pins pulled out from unidirectional (UD) laminate beam broken under crack-opening mode (mode I delamination fracture).

The procedure of manufacturing with Z-pins has recently become better known through publications.^[3] The manufacturing process used to make Z-pinned materials can be easily split in simplified individual steps. The manufacture of pin involves a pultrusion machine: a continuous-fiber tow is pulled through a bath of liquid resin and exits the bath through a die. The diameter of the pin (rodstock) is governed by the diameter of the die. Then a foam sandwich (perform), into which the pins are inserted, usually vertically, is located on top of the uncured laminate, directly above the area to be reinforced. The Z-pins are inserted into the laminates by the actions of the ultrasonic horn. The commercially available preforms are characterised further by the areal density of the contained reinforcement, which ranges from 0.5% to 7%.^[3]

The main objective of the work was to determine the mechanical properties of Z-pinned and unpinned composite samples evaluating Z-pins of two different

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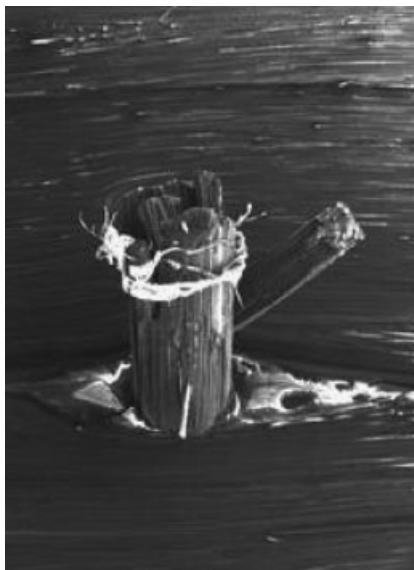


Figure 1.

E-SEM image of a Z-pin after mode I delamination fracture test. (Acceleration Voltage 10.0 kV, Spot 3.0, Magnification 39 \times , Detector secondary electron, Working Distance 9.5).

diameters: 0.28 and 0.5 mm; in each case the Z-pin areal density was 2%.

A small literature exists on the enhancement of mechanical properties due to different areal density of pins. Cartié and Partridge found that Z-pins gave a 6-fold to 25-fold increase in toughness as the areal density of pins was increased from 1% to 2%.^[4] No systematic studies have been found, however, on the knockdown of in-plane properties due to the presence of Z-pins. Nevertheless the thickness of Z-pinned samples is expected to increase as a function of the Z-pins density and consequently higher Z-pins density provide lower tensile moduli due to a local change in the fibre volume fraction with a fiber volume fraction dilution.

Experimental Part

Materials and Sample Analysis

The material investigated was a unidirectional thermoset polymer matrix composite

system constituted by matrix CYTEC 977-2 and fibre: carbon fibre yarn HTA – 12 k/35 with nominal ply thickness of 0.25 mm.

Composites plates have been prepared by laying up 10 carbon fiber/epoxy resin tapes with 58% nominal fiber volume. The uncured laid up laminates have been vacuum de-bulked and then pinned through the entire laminate depth using a hand-held ultrasonic gun. During insertion, the low-density foam holding the pins has been crushed. The pins have been shear cut at the surface of the laminate, and the excess pin length and the remaining foam have been discarded.

The composites plates have been reinforced with two different Z-pin diameters: 0.28 and 0.5 mm placed in different regions along the total nominal gage length, in each case at pin areal density of 2%. The Figure 2 shows two photos of a manufactured plate with the detail of the Z-pinned area. The inserted Z-pin is visible on the back face of the laminate and here the laminate thickness is greater as the insertion tends to make the laminate “swell” in the thickness direction.

Three laminate typology have been manufactured for the mechanical characterization: unidirectional laminates for the longitudinal and transversal tensile tests, bi-directional laminates [± 45]_{np} for the in-plane shear tests and unidirectional laminates with an artificial delamination created by means of a Teflon film insertion for the interlaminar fracture tests.

Before cutting the specimens from each plate, Non Destructive Inspection has been carried out to check the delivered laminate and also to ensure traceability of the specific specimen.

The Figure 3 shows a C-scan of a z-pinned panel. The z-pinning has been performed along the total nominal gage length and they are clearly recognizable. This happens because when the Ultrasound beam meets the z-pins it goes through a pure carbon fibre which has a different ultrasound impedance and then a different speed with respect to a carbon-resin system.

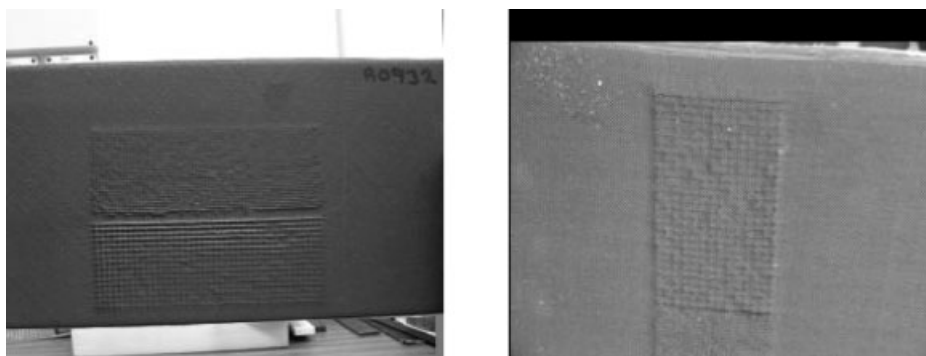


Figure 2.

Composite plate reinforced with two different Z-pin diameters: 0.28 and 0.5 mm.

Test Matrix Definition

Tensile and interlaminar fracture tests have been carried out and the following table reports the mechanical properties to be investigated.

The samples have been named with various labels related to the different Z-pin diameters: 0.28 mm (samples d_1), 0.5 mm (samples d_2) and unpinned (samples d).

Mechanical Test Equipment

A computer-aided electromechanical servo testing machine (type INSTRON 4505) has been used for all tests described in this paper. The load cell used for the tests was a 100 kN load cell, calibrated also within the range 0 kN to 10 kN. INSTRON Italy SIT Calibration Service performed the calibration for the

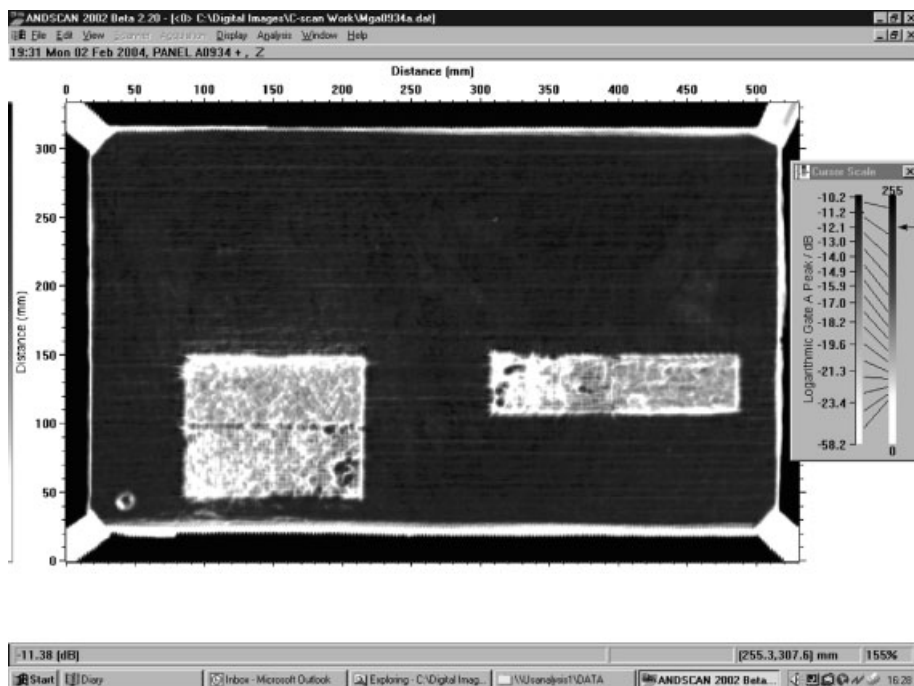


Figure 3.

C-scan showing z-pinned areas. Z-pins used were 2% density, 0.28 mm and 0.5 mm diameters.

Table 1.

Mechanical properties investigated during this test campaign.

E_1	LONGITUDINAL YOUNG'S MODULUS
σ_1^R	LONGITUDINAL TENSILE STRENGTH
E_2	TRANSVERSE YOUNG'S MODULUS
σ_2^R	TRANSVERSE TENSILE STRENGTH
G_{12}	IN-PLANE SHEAR MODULUS
τ_{12}	IN-PLANE SHEAR STRENGTH
DCB, G_I	FRACTURE TOUGHNESS FOR MODE I

load cell. The data acquisition software, SERIE IX Instron, has been used to record all the data signals.

In Plane Tensile Test Procedure

For the determination of the longitudinal, transversal and shear properties of unpinned and pinned specimens, tensile tests have been performed according to the standard ASTM D3039 and ASTM D3518.^[5]

Interlaminar Fracture Toughness Under MODE I Test Procedure

Interlaminar fracture toughness tests under mode I loading conditions has been carried out, according to the ASTM D 5528.^[5]

The location of the Z-pin area is reported schematically in Figure 4 with the indication of the inserted film position with respect to the specimen edge. The distance between the inserted film and the Z-pin area is about 30 mm. Hinged metallic tabs of about 1.0 mm thickness has been used as shown in figure. A release film e.g. PTFE oil of about 0.02 to 0.03 mm thickness

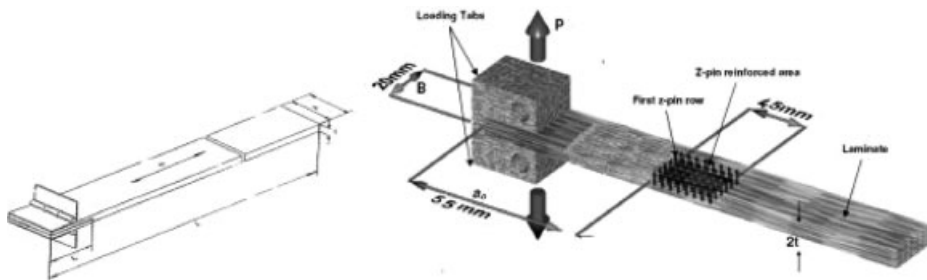
has been used for the introduction of the artificial delamination.

The Figure 5 shows a specimen used the interlaminar fracture test under mode I with the metallic tabs, before the test and during the test.

Measurements of delamination growth have been taken during the test from both sides of the specimen. A travelling optical device with a magnification of $10\times$ has been positioned on one side of the specimen and a mirror has been used to check for discrepancies from the two sides of the specimen. Crosshead separation may be used as a measure of opening displacement of the specimen provided that the deformation of the testing machine, with gripping fixture attached, is less than 2% of the opening displacement of the specimen.

An X-Y plotter has been used to record the load versus opening displacement during the test. The load-displacement data have been manually marked as the delamination front advances through each of the scale markings on the edge of the specimen.

The data acquired during the test are: the initial delamination length, $a_0 = 25$ mm (confirmed after the test by separating the two arms of the specimen by hand); the various delamination lengths, a (where $a = a_0 +$ the measured delamination length increments); the corresponding loads, P and displacements, δ . The initiation and propagation values of G_{IC} has been determined

**Figure 4.**

Typical sample geometry for an interlaminar fracture test (mode I) with piano hinges at one end and detail of the Z-pin location. The insert length is 25 mm, the distance between the insert film and the Z-pin area is 30 mm and the Z-pin area is 45 mm.

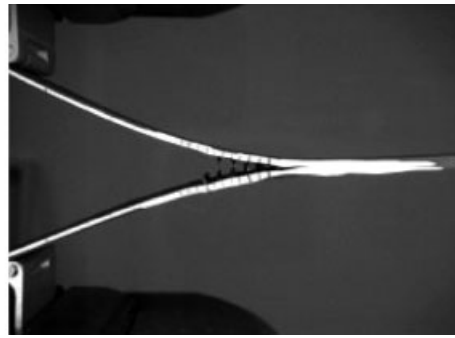


Figure 5.

Sample specimen for the interlaminar fracture test under mode I.

from the data acquired, generating a resistance curve R , by plotting the calculated G_{IC} versus crack length, a .

The modified beam theory (MBT) has been used in order to calculate the G_{IC} values. Following the modified beam theory (MBT) the energy release rate for a DCB specimen, in which the arms are considered to be clamped at the delamination front, is given by the expression:

$$G_{IC} = \frac{3P\delta}{2Ba} \quad (1)$$

By inserting into equation (1) the values of load, P , and displacement, δ , associated with growth at a particular crack length a , the critical energy release rate (G_{IC}) for that crack length can be determined.

However, in practice, the arms are not perfectly built-in and rotation may occur at the delamination front. This rotation effect may be accounted for by treating the DCB as if it contained a longer delamination at each length, $a + \Delta$, hence, the mode I fracture toughness using this modified beam theory is calculated from equation:

$$G_{IC} = \frac{3P\delta}{2b(a + \Delta)} \quad (2)$$

where b is the specimen width, and Δ is determined experimentally for each specimen, by plotting the cube root of the compliance, $C^{1/3}$, as a function of delamination length, a (the compliance is calculated as the ratio of displacement to the applied load, δ/P).

Results and Discussion

In-Plane Tensile Tests

The results in term of strength and the modulus of elasticity, calculated for all the tests, are reported in the Table 2, together with the average values and the standard deviation.

As shown in the above presented table, the effect of the orthogonal insertion of Z-pins is reduction of the in-plane properties, due to several combined effects:

- a local change in the fibre volume fraction with a fiber volume fraction dilution;
- the existence of resin-rich pockets around the Z-pins, causing stress concentrations;
- a possible misalignment of the longitudinal fibres due to the reinforcements insertion procedure;

These results are in agreement with the effects of inclusion of Z-pins over the stiffness and the strength of different laminate types described in.^[6]

The results point out the small reduction of shear modulus and of deformation capability due to the presence of the Z-pins. The effect on the in-plane shear stiffness was expected to be small, as deviated reinforcing fibers are capable to carry shear loads and hence to balance the added compliance of the resin-rich pockets. The drop off in the shear modulus, due to the presence of the Z-pins, is 9% in d_2 samples

Table 2.

Results of the in-plane tensile test.

Sample Typology	σ_{11} [MPa]	E_1 [GPa]	σ_{22} [MPa]	E_2 [GPa]	τ_{12} [MPa]	G_{12} [GPa]
Unpinned (<i>d</i>)	2059 ± 88	146.7 ± 7.8	55.45 ± 7.42	9.89 ± 0.11	74.10 ± 2.01	5.65 ± 0.03
Z-pinned 0.28 mm (<i>d</i> ₁)	1439 ± 79	140.8 ± 8.8	50.20 ± 2.01	9.82 ± 0.11	70.63 ± 2.32	4.98 ± 0.18
Z-pinned 0.5 mm (<i>d</i> ₂)	1189 ± 96	127.3 ± 7.7	42.06 ± 3.80	9.73 ± 0.12	70.90 ± 0.85	5.16 ± 0.10

and 12% in d_1 samples; the drop off in the shear stress is 4% in both cases.

Failure occurred within the gauge length for the most tensile specimens. Failure in the grips occurred in only two out of a total of seven specimens tested in the transversal direction, eliminating them from the results.

Interlaminar Fracture Toughness Under MODE I Tests

The load-displacement data for the Z-pinned specimens are presented in Figure 6.

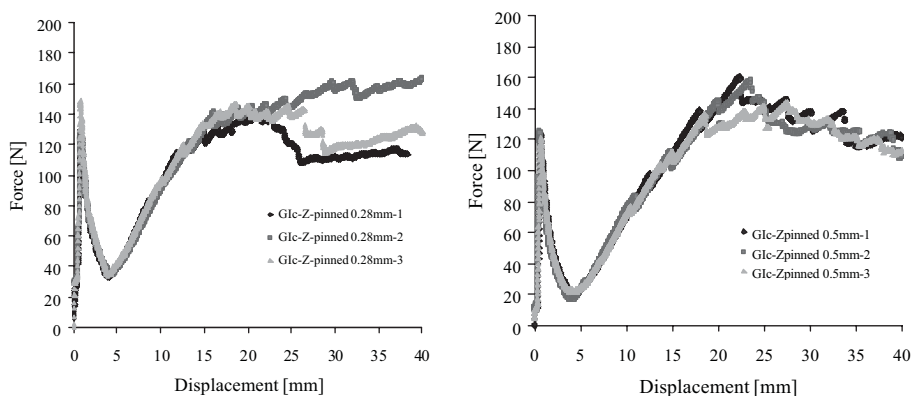
Crack initiation appears to be unaffected by the presence of the pins up to a cross-head displacement of 5 mm. At this displacement the crack propagates to the Z-pinned area and the load increase significantly. The load-displacement curve for the Z-pinned samples exhibits very stable crack characteristics right up to a displacement value of 15 to 20 mm. At this displacement, the crack front passes through the middle of the pinned region of the specimen ($a=80$ mm). The subse-

quent load drops indicate the pulling out of the pins; catastrophic failure follows.

Initiation and propagation values of G_{IC} has been determined from the data acquired, and these values have been used to generate a resistance curve, by plotting the calculated G_{IC} versus the crack length, a , as shown in the Figure 7.

The resistance curves for the Z-pinned samples show a behaviour similar to the unpinned samples one up to crack length $a=55$ mm, corresponding to the unpinned area. Here, up to this crack length, the crack initiation appears to be unaffected by the presence of the pins; then an expected significant increase in the resistance to delamination cracking occurs.

In the Z-pinned area the crack growth is unstable and it is difficult to take the plateau values as a good indicator of the crack propagation resistance of these specimens. It is reasonable to take a value of about 4000 ± 500 J/m² for the interlaminar fracture toughness of samples with z-pins diameter = 0.28 mm and a value of about 2700 ± 300 J/m² for the samples with

**Figure 6.**

Load-displacement data for samples with 0.28 diameter Z-pins (on the left) and 0.5 diameter Z-pins (on the right), tested in mode I loading conditions.

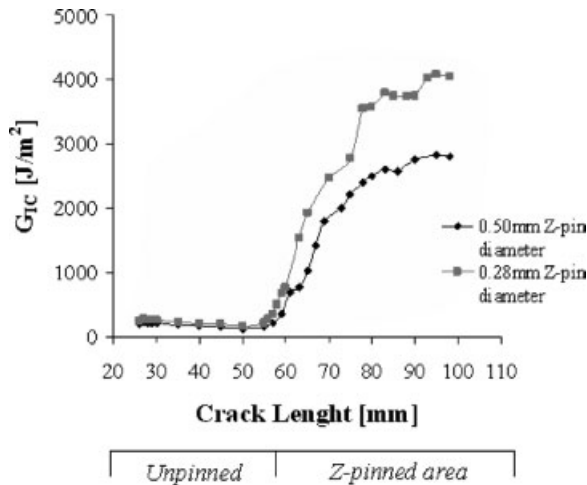


Figure 7.

R curve for the samples for samples with 0.28 mm Z-pin diameter and 0.5 mm Z-pin diameter, tested in mode I loading conditions.

z-pins diameter = 0.5 mm. The d_1 samples (0.28 mm z-pin diameter) seem to offer better performances in term of interlaminar fracture toughness and this result is in agreement with the FE prediction on the effect of Z-pin diameter on mode I delamination resistance for areal density of pinning of 2%.^[7] For fixed Z-pin densities, the force required to continue crack propagation is higher for Z-pins of smaller diameter. Indeed, the mechanism that generates the Z-pin reaction force during pullout is based on friction and the frictional areas are inversely proportional to the Z-pin diameter. In practice the apparent toughness is not exactly proportional to the Z-pin diameter due to the combined effects of bending of the arms of the beam and elastic deformation of the Z-pins.

Conclusions

This paper describes the results achieved during the testing campaign performed on Z-pinned and unpinned composite samples. Tensile and interlaminar fracture tests under mode I have been carried out on

thermoset polymer matrix composite samples made of CYTEC 977-2 and carbon fibre yarn HTA-12K/35. The presence of the Z-pins has been found to significantly increase the interlaminar fracture toughness. The crack resistance improvement has been found to be accompanied by a reduction of the in-plane stiffness properties for a Z-pins density of 2%.

Finally the effect of Z-pin diameter at a fixed areal density has been highlighted: the 0.28 mm reinforcements seem to offer better performances in term of in-plane properties and delamination resistance.

Acknowledgements: This work has been performed within the frame of the EUCLID project Jp 3.29, DAMOCLES II.

A special acknowledgment is due to Cinzia Toscano of the Non destructive Testing Laboratory at CIRA for her precious help with the C-scan analyses.

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