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Time Storage Effect of the Resin on the Toughness of a Unidirectional Carbon Fibre

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Abstract: In the present study we investigate the effect of the time storage of the epoxy resin on the interlaminar fracture toughness of a three unidirectional carbon fibre. The interlaminar fracture behaviour was studied using the mode I, Double Cantilever Beam (DCB) test, the mode II, End Notched Flexure (ENF) test and a mixed mode bending (I/II) in order to determine the energy required for the initiation and growth of an artificial crack. The materials were made by a 32 stacking of unidirectional prepared carbon fibre. The matrix used was an epoxy of type (TGDMA) without a modification. The delamination energies of these three materials were compared in orders to characterize their mechanical properties.

Key words: Mode I (DCB), mode II (ENF), mixed mode bending (I/II), matrix epoxy, unidirectional carbon fiber, strain energy rate

INTRODUCTION

Fracture mechanics has found extensive applications in damage analysis of composite laminates, especially in delamination (Bathias and Laksimi, 1985; Bolotin, 2001). One of the most important parameters in the application of fracture mechanics in composite structures is the strain energy release rate. Composite materials offer some exciting advantages over more traditional metallic materials. Applications range from ski sticks, tennis rackets, parts, in biomedical implants and reinforcement of highway bridges to advanced aircraft and space vehicles. More widely diverse applications suffer from difficulties in recycling to question of long term durability and the inability to accurately predict the life. One of the obstacles hindering the acceptance of polymer composites in civil engineering applications is the susceptibility of the polymeric matrix to degradation initiated by moisture, temperature and corrosive chemical environments. Composites offer excellent corrosion resistance to environmental agents as well as the advantages of high stiffness-to weight and strength-to-weight ratios when compared to conventional construction materials. Delamination growth represents a critical failure mode in laminated composite structures. A typical procedure for assessing the property for a delamination to grow is to compare the energy release rate, G , to its critical value or toughness, G_c (Hull and Clyne, 1987). Since G_c depend on the type of loading, the Energy Release Rate (ERR) is typically decomposed into individual components and the G is compared to the value of G_c . Various techniques

have been successfully employed to suppress and or to delay delamination (Wang and Zhao, 1995). It has been noted that delamination in a composite laminate usually occurs at the interface of different oriented plies. The mode of delamination failure depends on the external loading conditions and on the intrinsic properties of fibre and resin. The interlaminar fracture behaviour is one of the most important characteristics related to the overall performance of the composite system (Bruner *et al.*, 1994 ; Flüeler and Brunner, 1992). In service, most failures occur by mixed mode delamination cracks. The Mixed Mode Bending test has been developed to produce a wide range of mixed-mode conditions for composite materials specimens (Bruner *et al.*, 1994; Benzeggagh and Kennane, 1996; Hashemi *et al.*, 1989, 1999).

The study reported here was carried out to investigate the effect of the time storage of the resin on the mechanical behaviour of a three unidirectional prepreg carbon fibres. The matrix epoxy was used without a modification. Double Cantilever Beam (DCB), End Notched Flexure (ENF) and a mixed mode bending (I/II) were carried out in order to analyze the toughness and failure mechanisms, also in each mode of test two different theories have been used in order to determinate which would be the most appropriate.

MATERIALS AND METHODS

Three composites materials have been used in the present investigation. The materials were denominated by 3 M, 4 N and 4 C. The materials were made by a stacking

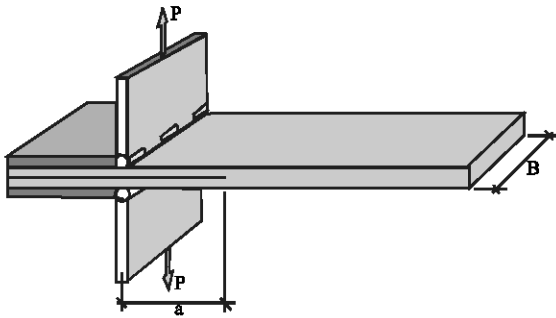


Fig. 1: Mode I, DCB test

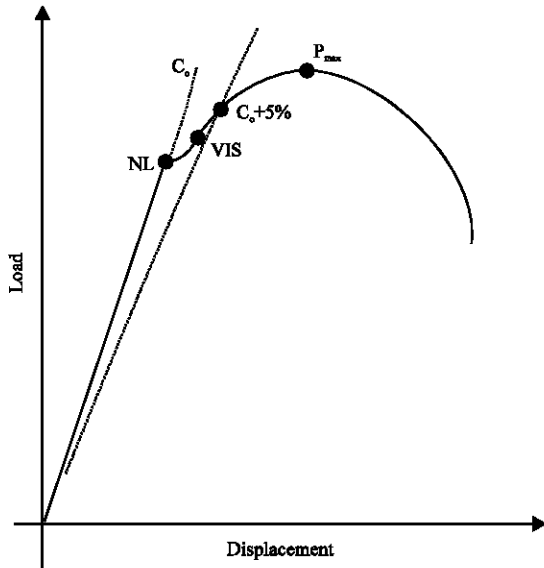


Fig. 2: Load-displacement curve

of unidirectional prepared carbon fibre. The matrix was an epoxy of type (TGMDA) without a modification reinforced with 33% in weight of carbon fibre.

The time storage of the resin for the materials 4 C, 3 M and 4 N was 4, 7 and 8 months, respectively.

They were manufactured panels of 350 by 350 mm, with a nominal thickness of 5.5 mm, made of 32 prepared layers of sequences $(0)_{16s}$. During the processing, a thin film of polyamide of 20 μm thickness was introduced in the middle of the plane in order to simulate an artificial crack.

Mode I interlaminar test: Five DCB specimens (Hojo *et al.*, 1995; Kalbermatten *et al.*, 1992; Naik *et al.*, 1991; O'Brien and Martin, 1993) from each composite laminate, measuring $120 \times 20 \times 5.5$ mm with an insert length of 60 mm, were tested under ambient conditions using an Instron machine with a 1 kN load cell according to ASTM D5528 (ASTM D 5528). A pair of metallic hinges was glued to the loading end of the specimen in order to

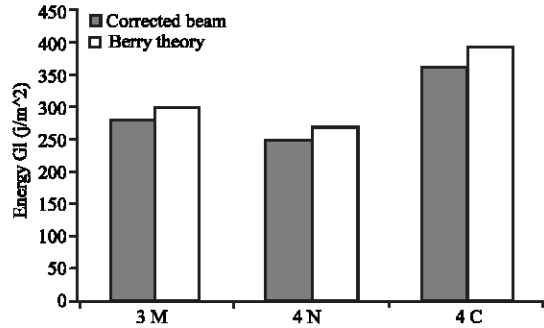


Fig. 3: Energy values G_{IC} for the three materials calculated at the maximum load

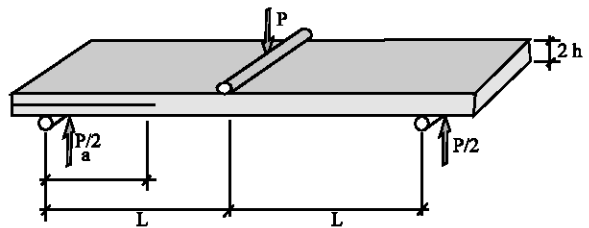


Fig. 4: Energy values G_{IC} for the three materials calculated at point 5%

enable the load to be applied (Fig.1). The tests were carried out at a constant loading rate of 0.5 mm min^{-1} . The critical values of the energy of the delamination, G_{IC} , were calculated at the characteristic points (NL, 5% and P_{max}) (Fig. 2).

The crack propagation energy values were calculated using the corrected beam theory.

$$G_{IC} = \frac{3P\delta}{2B(a + |\Delta|)} \quad (1)$$

Where P is the force, δ the displacement of the notch lip, the crack length and B the specimen width. The corrected beam theory requires the determination of a correction factor Δ , which takes into account crack tip rotation and shear deformation. This factor Δ was obtained by drawing the third power compliance against crack length ($C^{1/3}$ -a). In addition, the experimental compliance calibration or Berry's method was employed in this case.

$$G_{IC} = \frac{nP\delta}{2Ba} \quad (2)$$

Where n is the slope of the plot $\log C - \log a$ ($C = Ka^n$).

Figure 3 and 4 presents the medium values for five specimens of the crack propagation energy G_{IC} , for the three composite materials according to the

Table 1: Energy G_{Ic} at NL point

Materials	3 M	4 N	4 C
E_{11} (MPa)	140000	139500	141000
E_{22} (MPa)	9000	8500	9600
G_{13} (MPa)	5000	3700	5500
c (mm)	69	69	68.5

Table 2: Energy G_{Ic} at N.L. point

Materials	Corrected beam G_{IIC} ($J m^{-2}$)	Experimental calibration G_{IIC} ($J m^{-2}$)
3M	1223.99	719.94
4N	1090	650.72
4C	1431.4	790.38

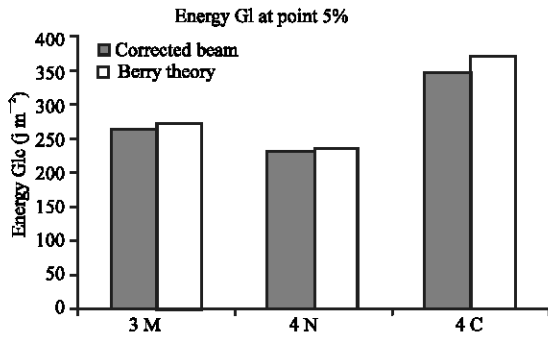


Fig. 5: Mode II, ENF test

corrected Beam and Berry's theories at the maximum load and 5% point. From the Fig. 3, it can be seen that the corrected beam theory had provided more conservative values of the energy. The material 4 C had the highest fracture energy about ($355 J m^{-2}$), while the others two composites presented quite similar energy values (the energy of 3 M ($280 J m^{-2}$) was slightly superior to 4 N). In Table 1, we present the medium values of G_{Ic} at NL point.

Mode II interlaminar test: For the mode II test, an End Notched Flexural (ENF) specimen geometry was used (Akay *et al.*, 1995; Carlsson and Gillespie, 1989). The specimen was loaded in a standard three point bending fixture at a crosshead speed of $0.5 mm min^{-1}$ (Davies, 1993) (Fig. 5). The delamination energy G_{IIC} was calculated at the characteristic points according to the corrected beam theory:

$$G_{IIC} = \frac{9P^2a^2}{16B^2Eh^3} \quad (3)$$

The value of E is the modulus measured during the compliance calibration for $a = 0$. It is calculated as:

$$E = \frac{L^3}{4BCh^3} \quad (4)$$

In this mode, according to the experimental compliance calibration method:

$$G_{IIC} = \frac{3mP^2a^2}{2B} \quad (5)$$

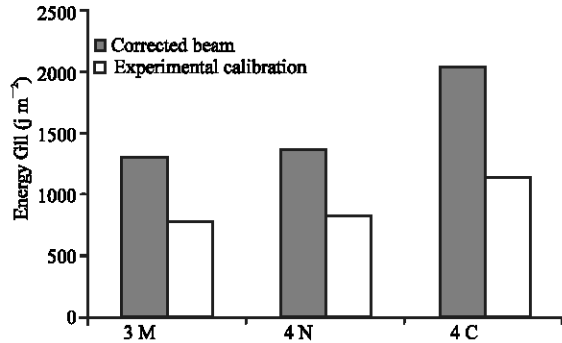


Fig. 6: Energy values G_{IIC} for the three materials calculated at the maximum load

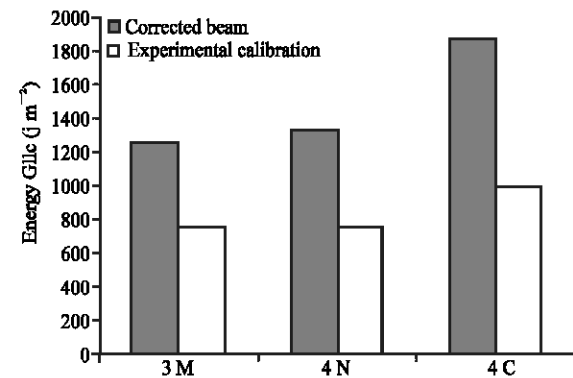


Fig. 7: Energy values G_{IIC} for the three materials calculated at point 5%

After the fracture tests, the specimens are broken open. The distances from the marks made during the calibration load cycles to the tip of the inserted film are measured and the mean of the values at the edges and the centre of the specimen are plotted against the corresponding compliances. A least square linear regression is then carried out, of the form:

$$C = C_0 + ma^3 \quad (6)$$

The value of the crack length can thus be obtained; m being the slope of the curve $C = f(a^3)$ and C_0 the value of C at the origin.

Figure 6 and 7 represents the critical energy values of G_{IIC} of the delamination at the maximum load and 5% point for the three composite materials using the corrected beam theory and the experimental calibration method. The

material 4 C present the highest resistance to the delamination about (1150 J m⁻²). The other two composites (3 M and 4 N) have more or less similar fracture energy (750 J m⁻²).

The values of the crack energy were more conservative using the experimental calibration method. The values of G_{IIc} at the NL point are presented in Table 2.

Mixed mode bending (I/II) test: A mixed mode delamination test procedure was developed combining Double Cantilever Beam (DCB) mode I loading and End-notch Fixture (ENF) mode II loading. By loading with a lever, a single applied load simultaneously produces mode I and mode II bending loads on the specimen. In order to carry out the mixed mode bending, a device was designed, Fig. 8. The Table 3 shows the mechanical properties of the three composite materials.

For a DCB specimen:

$$G_I = \frac{12a^2P_1^2}{B^2h^3E_{11}} \quad (7)$$

$$P_1 = \left(\frac{3c-L}{4L} \right) P \quad (8)$$

Where:

P₁: Load for mode I

P₂: Load for mode II

E₁₁: Longitudinal young modulus

Table 3: Mechanical properties

G13 (MPa)	5000	3700	5500
c(mm)	69	69	68.5
L(mm)	50	50	50

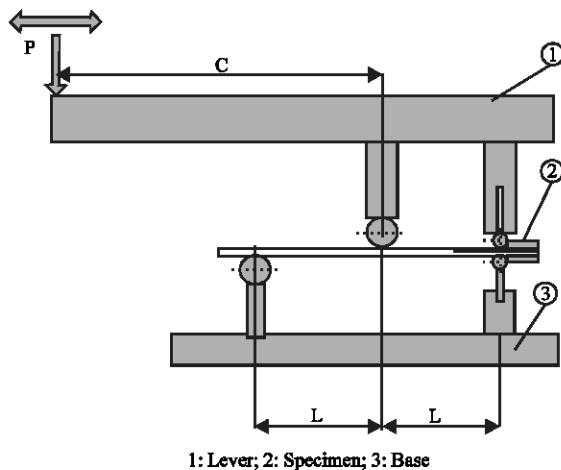


Fig. 8: Mixed mode bending

For an ENF specimen:

$$G_{II} = \frac{9a^2P_2^2}{16B^2h^2E_{11}} \quad (9)$$

The energy G_I and G_{II} depend on the distance c. A linear analysis based on the beam theory has been developed by Reeder and Crews (1992), the contribution of mode I and mode II to the critical total strain energy release rate is given by the following relation:

$$G_c = G_I + G_{II} = \frac{3a^2P^2}{16B^2h^3L^2E_{11}} \left[4(3c-L)^2 + 3(c+L)^2 \right] \quad (10)$$

The Fig. 9 and 10 presents the medium values of the critical energy, G_I, G_{II} and G_c of the delamination for the three composite materials. Starting from the Fig. 9, it can be noted that the material 4 C had the greater energies (G_I, G_{II}, G_c) to the delamination (500 J m⁻²). The energy of

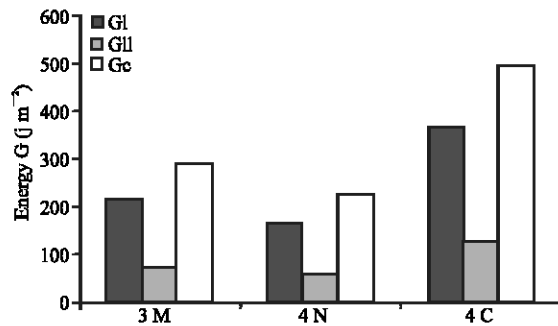


Fig. 9: Energy values G_c for the three materials calculated at the maximum load

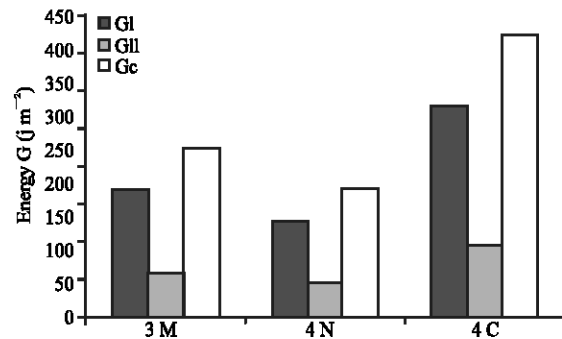


Fig. 10: Energy values G_c for the three materials calculated at point 5%

Table 4: Energy G_c at NL point

Materials	G _I J m ⁻²	G _{II} J m ⁻²	G _c J m ⁻²
3M	157.77	54.94	212.71
4N	126.85	43.87	170.72
4C	291.71	100.14	391.85

the crack toughness of the material 3 M was slightly higher to the 4 N. Also the Table 4 showed the medium values of the fracture energy at NL point.

DISCUSSION

In mode I: For the three tested materials, the most resistant to the delamination growth in mode I was the material denominated 4 C, presenting a release rate energy, approximately about 30% higher than the others two materials. The materials 3 M and 4 N offer a very similar resistance (slightly greater 3 M), in fact, after the realization of the tests, the manufacturer of the specimens had confirmed that both materials were the same one, with the only difference of which in the case of 4 N, the time of storage of the resin recommended by the manufacturer had been exceeded. From the two theories used to calculate the energy G_I , the corrected beam theory was chosen (more conservative values).

In mode II: From the three composites tested, the material 4 C present a higher resistance to the delamination in mode II. The materials 3 M and 4 N showed an identical behaviour. The experimental calibration method was used to calculate the energy release rate G_{II} . This method had given more conservative values of the energy release rate.

In mixed mode bending: In mixed mode, the material 4 C present the energies (G_I , G_{II} and $G_{I/II}$) of delamination superiors than the others two materials. The materials 3M and 4 N have the same behaviour observed in mode I and mode II; the material 3 M present values of G_I slightly greater. The material 3 M present higher values of the energy G_c .

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