THE EFFECT OF MOISTURE ON THE INTERLAMINAR FRACTURE TOUGHNESS OF CFRP LAMINATE

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Keywords: *interlaminar fracture toughness, laminate, energy release rate*

Abstract

The effect of moisture on the mode I and mode II interlaminar fracture toughness of carbon fibre/epoxy laminates was studied experimentally with DCB (Double Cantilever Beam) and ENF (End Notched Flexure) tests. Unidirectional $[0°]_{24}$ *lay-up was used in the study. The material used in the specimens was AS4/3501-6 unidirectional prepreg tape. Three types of specimens were tested: 1) dry specimens, 2) environmentally exposed specimens, and 3) specimens at ambient humidity.*

The tests were performed with a servo-hydraulic test machine. A 0.5 kN load cell was used in the DCB tests. The DCB test procedure included two loading cycles making it possible to measure two separate crack initiation values and several crack propagation values. The ENF tests were performed with a typical three point bending jig.

A higher mode I crack initiation value was obtained for an environmentally exposed (wet) laminate when interlaminar fracture toughness was measured from the delamination insert. When measured from the precrack, the crack initiation values showed lower dependence on moisture. The delamination-resistance curves were similar for all three types of specimens. Mode II values increased slightly with moisture.

Increase in the insert crack initiation toughness was most likely caused by moisture related toughening of the resin-rich area at the insert tip.

1 Introduction

One of the frequent failure modes of composite laminates is delamination. Delamination is an interface failure between the laminate layers. Delamination failures in laminates can be divided into two groups: manufacturing induced delaminations and service induced delaminations.

Delamination may be introduced for example during processing of the composite lay-up. In service, impacts and free-edge stresses are common causes of delaminations. Typically delamination causes degradation in laminate stiffness and strength. The propagation of the delamination can lead to a catastrophic failure of the structure without visibly detectable signs.

Delamination and debonding are crack-like discontinuities in the structure. Thus, fracture mechanics can be used in the analysis. Common practice is to investigate interlaminar fracture toughness using the strain energy release rate *G*.

Many tests have been introduced for measuring the interlaminar fracture toughness. The most common test methods DCB (Double Cantilever Beam) and ENF (End Notched Flexure) use beam-like specimens with artificial delaminations. Experimentally measured energy release rate values can be utilized in the delamination analyses.

Mechanical properties of carbon fibre/epoxy laminates are typically significantly affected by moisture absorption. The interlaminar fracture toughness is dependent on different laminate properties such as matrix toughness and the strength of fibre/matrix interfacial bond. The effects of moisture on the interlaminar fracture toughness have rarely been investigated in the literature. Moisture effects on these properties and the resulting changes in the critical energy release rates were evaluated in the study.

2 Test methods

There are three standardized test methods for the determination of the interlaminar fracture toughness of fibre-reinforced composite laminates. All standardized methods have been limited to symmetric $[0°]_n$ unidirectional laminates.

Double Cantilever Beam (DCB) test is a standard test method for determining the interlaminar fracture toughness G_C for the crack opening mode I. End Notched Flexure (ENF) method is for the crack shearing mode II testing and Mixed-Mode Bending (MMB) is for the mixed-mode I/II testing.

The energy release rate G is the loss of energy dU in the test specimen per unit of specimen width b for an infinitesimal increase in delamination length da for delamination growing under a constant displacement. Therefore, energy release rate can be expressed with equation

$$G = -\frac{1}{b}\frac{dU}{da}. \qquad (1)$$

Interlaminar fracture toughness tests DCB and ENF use edge-cracked specimens. Energy release rates can be calculated from the test data containing simultaneously measured values of force P, displacement δ, and crack length a. Typically equation (1) is expressed in a form

$$G = \frac{1}{2b}\frac{dC}{da}, \qquad (2)$$

where compliance $C= \delta/P$, and G for the cracked specimen can be calculated from the compliance change dC per increase in the delamination length da.

2.1 DCB test

Double cantilever beam specimens were used to determine the critical energy release rate G_{IC}. The test method produces initiation and subsequent propagation values of G_{IC} for mode I crack opening fracture toughness. A delamination-resistance curve can be generated by plotting G_{IC} values as a function of crack length a. Geometry for the DCB specimen is shown in Fig 1.

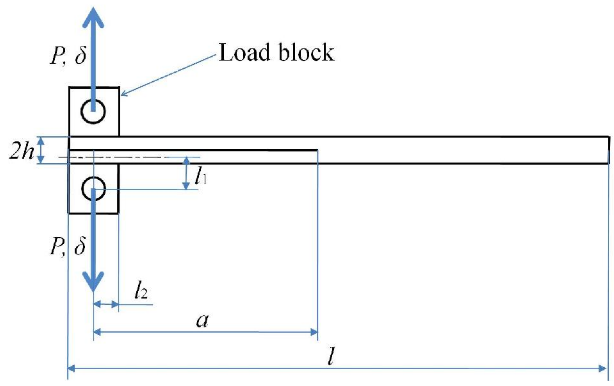
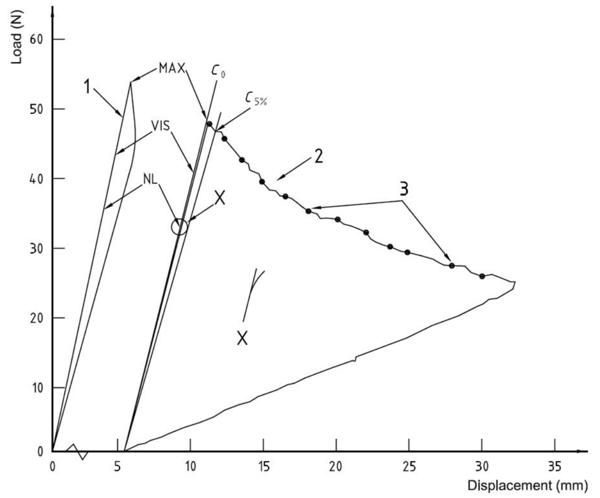


Fig. 1. Geometry for the DCB specimen

Crack-opening load is applied to the test specimen through load blocks. Blocks are attached to the cracked end of the specimens with adhesive. DCB tests are made under displacement control at constant displacement rate. Several delamination initiation G_{IC} values can be obtained from the test. These include values determined 1) from the point of deviation from linearity in the load-displacement curve (NL), 2) from the point at which delamination is visually observed on the edge of the specimen (VIS), and 3) at the point where the compliance has increased 5 % or the load has reached the maximum value ($C_{5\ \%}$/ MAX).

Delamination initiation G_{IC} values are obtained in the first loading cycle from the delamination insert and in the second loading cycle from the precrack. In addition, crack propagation G_{IC} values are determined from the second loading cycle. The load-displacement curve of the DCB test is illustrated in Fig. 2.



1	Crack initiation followed by unloading
2	Crack propagation
3	Crack propagation markers
C_0	Compliance (linear part, 2nd loading cycle)
$C_{5\%}$	5 % increase in compliance
MAX	Maximum load point
NL	Non-linearity point
VIS	Visual point

Fig. 2. Load-displacement curve for DCB test [1]

The figure shows initiation loading from the insert (1) followed by re-initiation and crack propagation (2). Also crack propagation markers (3) are denoted in the figure. Note that reload curve has been offset 5 mm for clarity.

2.1.1 DCB test data reduction

In this study DCB test data reduction was done with the Corrected Beam Theory (CBT) method. The relation between the delamination length and the compliance is established by plotting the cube-root of the compliance $(C/N)^{1/3}$ (corrected with the load block correction *N*) as a function of delamination length *a*.

The corrected beam theory analysis corrects the assumption of simple bending for the transverse shear and for deformation beyond the crack tip. This is achieved by the addition of the correction Δ to the crack length. The critical energy release rate can be calculated from equation [1]

$$G_{IC} = \frac{3P\delta}{2b(a+\Delta)} \cdot \frac{F}{N}, \qquad (3)$$

where *F* is the large-displacement correction

$$F = 1 - \frac{3}{10}\left(\frac{\delta}{a}\right)^2 - \frac{2}{3}\left(\frac{\delta\, l_1}{a^2}\right), \qquad (4)$$

and *N* the load block correction

$$N = 1 - \left(\frac{l_2}{a}\right)^3 - \frac{9}{8}\left[1 - \left(\frac{l_2}{a}\right)^2\right]\frac{\delta\, l_1}{a^2} - \frac{9}{35}\left(\frac{\delta}{a}\right)^2. \qquad (5)$$

Dimensions l_1 and l_2 in equations (4) and (5) are defined in Fig. 1.

2.2 ENF test

End notched flexure test method is used to determine the critical crack shearing energy release rate G_{IIC}. The ENF test method produces a crack initiation G_{IIC} value which is measured from the delamination insert. Typically ENF test is unstable, hence crack propagation G_{IIC} values cannot be measured.

Precracking of the ENF specimen can be done in mode I loading. However, mode I load is likely to create the fibre-bridging which is undesirable. A precrack could also be induced by loading the specimen in a stable crack length regime, $a>0.7l$ (Fig. 3), until a short crack propagation is achieved. Unfortunately, it is difficult to detect the exact position of crack tip and obtain uniform crack front shape. Therefore, tests are done without precracking and G_{IIC} values are determined from the delamination insert. [2]

The shear loading is applied to the test specimen with a three-point bending jig under displacement control at constant displacement rate. In the ENF test delamination initiation G_{IIC} values are determined from the non-linearity point (NL), visual point (VIS) or from the $C_{5\%}$/MAX point. Geometry for the ENF specimen is shown in Fig 3.

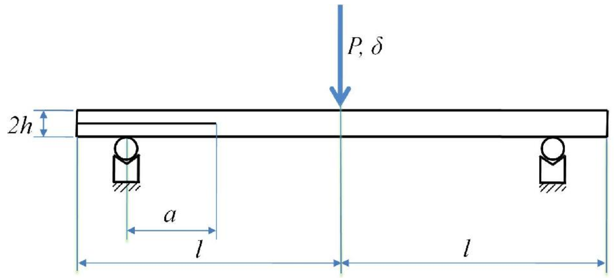


Fig. 3. Geometry for the ENF specimen

2.2.1 ENF test data reduction

In this study ENF test data reduction was done with the corrected beam theory method proposed by Wang and Williams [3]. The critical energy release rate according to the CBT method is

$$G_{IIC} = \frac{9P^2(a+0.42\chi)^2}{16b^2h^3E_1}, \quad (6)$$

where the term 0.42χ is a crack length correction that accounts for deflection and rotation at the crack tip. The correction is needed because the beam theory assumes that the compliance at the crack root is zero. The factor χ can be given as

$$\chi = h\sqrt{\frac{E_1}{11G_{13}}\left[3-2\left(\frac{\Gamma}{1+\Gamma}\right)^2\right]}, \quad (7)$$

where the transverse modulus correction parameter is

$$\Gamma = 1.18\frac{\sqrt{E_1E_2}}{G_{13}}. \quad (8)$$

3 Test arrangements

DCB tests were performed according to the International Organization for Standardization ISO 15024 test standard for the determination of mode I interlaminar fracture toughness [1]. For ENF tests the Japanese Industrial Standard JIS K 7086 was used as a reference [4]. In the test instrumentation design the preliminary research done by the author was utilized [5].

3.1 Test specimens

Dimensions of the DCB test specimens were 20×125 mm with a 64 mm artificial delamination. For ENF specimens the dimensions used were 20×120 mm with a 35 mm delamination. Unidirectional $[0°]_{24}$ lay-up led to the laminate thickness close to 3.5 mm for the specimens.

3.2 Specimen manufacture

The specimens were manufactured from the AS4/3501-6 carbon fibre/epoxy prepreg. The measured ply properties of the material are presented in Table 1. In equations (7) and (8), the interlaminar shear modulus G_{13} is required. Because G_{13} is unknown, the in-plane shear modulus, G_{12}, is used as an approximation to G_{13} of the unidirectional test specimen.

Table 1. AS4/3501-6 ply properties

E_1 (GPa)	140
E_2 (GPa)	10
G_{12} (GPa)	5.17
V_{12}	0.3
t (mm)	0.146

Laminates were laid up onto a flat aluminum mold. Debulking was performed after five layers during the lay-up. Two 340×340 mm panels were manufactured. A non-adhesive FEP (Fluorinated Ethylene Propylene) insert film with thickness less than 13 μm was placed at the laminate mid-plane to simulate a sharp delamination crack.

The panels were cured in an autoclave. The cure temperature was 177°C and the curing time 2 hours. The cure cycle included a dwell period of 1 hour at 120°C. The autoclave pressure was 5.5 bars. Theoretical fibre volume content of the laminates manufactured with the described procedure is 59-60 %.

The test specimens were machined from the panels using a diamond blade band sawing machine. Fifteen DCB and fifteen ENF specimens were machined from the panels. Specimen edges were sanded with very fine P240 sandpaper and polished with ultrafine P2000 sandpaper.

Aluminum load blocks were bonded to the DCB specimens with Scotch-Weld DP-190 paste adhesive. A thin layer of typewriter correction fluid was applied to the specimen edges to aid the observation of delamination growth. Also delamination length marks were drawn along the edge of the specimens for the crack length measurements. DCB and ENF test specimens are illustrated in Fig. 4. and Fig. 5.

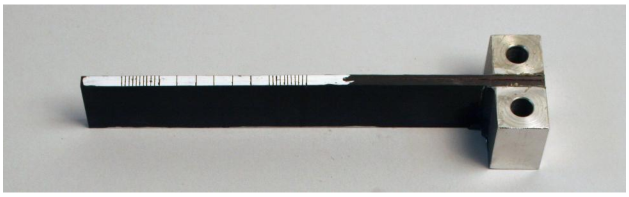


Fig. 4. DCB test specimen

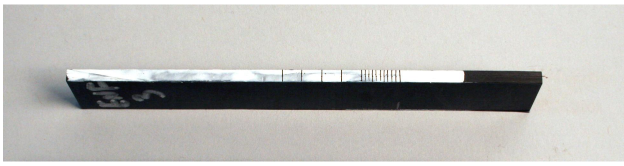


Fig. 5. ENF test specimen

3.3 Specimen conditioning

Three different types of specimens were used in the study: 1) dry specimens, 2) environmentally exposed specimens, and 3) specimens at ambient humidity. The specimens of first type were dried 7 days in an autoclave (90 °C, -0.8 bar). The specimens of second type were exposed 8 days in the weather chamber (60 °C, 95 %RH). The specimens of third type were stored at room temperature at ambient humidity (23 ± 3 °C, 50 ±10 %RH). The test matrix is presented in Table 2.

Table 2. The test matrix

Specimen type	Lay-up	Exposure	Number of specimens
DCB	$(0°)_{24}$	23 ± 3 °C, 50 ±10 %RH	5
DCB	$(0°)_{24}$	60 °C, 95 %RH	5
DCB	$(0°)_{24}$	90 °C, -0.8 bar	5
ENF	$(0°)_{24}$	23 ± 3 °C, 50 ±10 %RH	5
ENF	$(0°)_{24}$	60 °C, 95 %RH	5
ENF	$(0°)_{24}$	90 °C, -0.8 bar	5
		TOTAL	30

3.4 Test equipment

The tests were performed with a servo-hydraulic test machine. During the tests the test machine cross-head displacement δ and corresponding load P were measured and recorded with MTS software. A travelling optical microscope was used for measuring the delamination lengths in DCB tests. Additional 0.5 kN load cell was used with DCB specimens for measuring the low loads typically met in the tests. The test equipment included also a signal button which was used for identifying the displacement and load values for each corresponding delamination lengths. The button signal was also recorded to the test data. Test instrumentation is presented in Fig. 6.

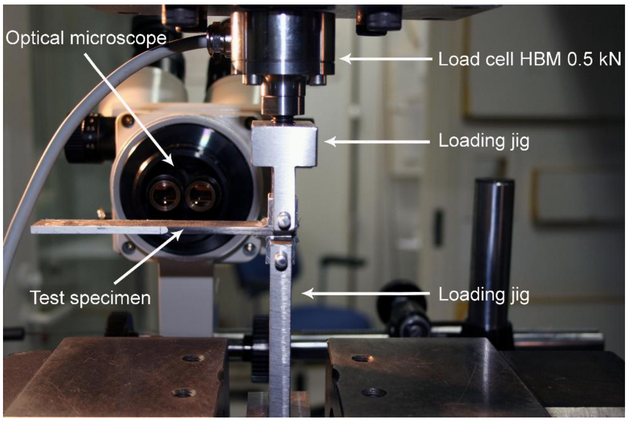


Fig. 6. DCB test instrumentation

3.5 Test procedure

The crack opening load was applied through the load block of the DCB specimen with constant displacement 2 mm/min. The DCB test procedure included two loading cycles making it possible to measure two separate crack initiation values and several crack propagation values. Propagation lengths were measured from the marks drawn along the edge of the specimens. The signal button was applied when crack front was visually observed to propagate past length marks.

ENF tests were performed with a typical three point bending jig with the test speed of 0.5 mm/min. All tests were performed at room temperature and ambient humidity.

All dried and environmentally exposed specimens were allowed to cool down to room temperature before testing.

4 Test results

The test results presented in this chapter include energy release rate charts from the DCB and ENF tests. The values are determined from the load-displacement curve non-linearity (NL) point. Indicated by the physical evidence from the previous studies it has been suggested that these values correspond to the physical onset of the delamination in the interior of the specimen width [6]. The DCB test results include also delamination-resistance curves (R-curves) for each test series. All energy release rate values are average values of five specimens.

4.1 DCB test results

Fig. 7 and Fig. 8 summarize the test results for the DCB specimens. In the charts the test series which is environmentally exposed is designated as “Wet”. The test series which was dried in an autoclave is designated as “Dry” and the series kept in ambient humidity as “Room”. The critical energy release rate G_{IC} values measured from the delamination insert are illustrated in Fig. 7 whereas Fig. 8 summarizes the G_{IC} precrack values.

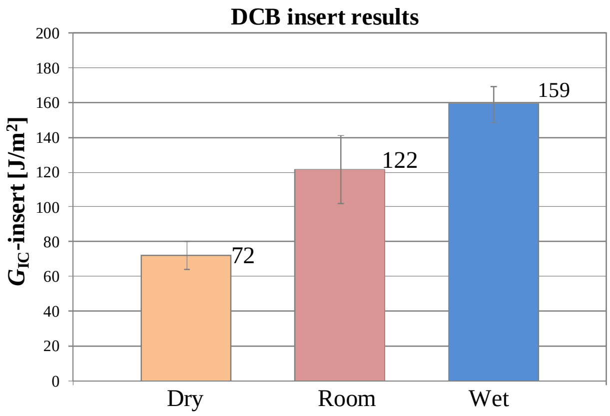


Fig. 7. DCB insert results

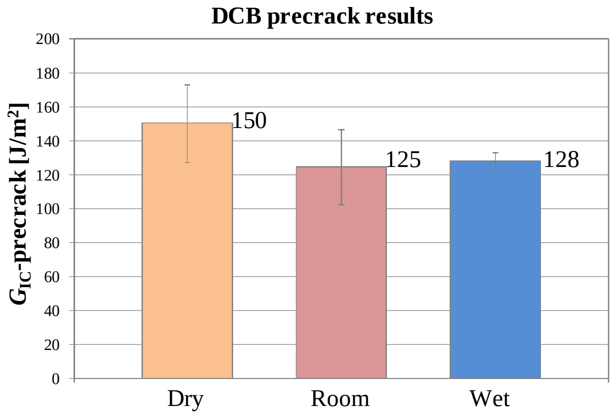


Fig. 8. DCB precrack results

For the crack opening mode I, a higher crack initiation value was obtained for the environmentally exposed test series when interlaminar fracture toughness was measured from the delamination insert (Fig. 7). When measured from the precrack, the average G value showed less dependence on moisture content (Fig. 8).

The crack initiation value depended whether the value had been measured from the insert or from the precrack. The most significant difference was found for the dry test series which yielded crack initiation insert and precrack values 72 J/m^2 and 150 J/m^2, respectively. The wet test series produced a higher insert value 159 J/m^2 and a lower precrack value 128 J/m^2. Insert and precrack values were nearly equal for the room condition test series.

Fig. 9 presents delamination-resistance curves which are generated by plotting G_{IC} values as a function of crack length a-a_0. Symbol a refers to total crack length and a_0 to the length of the delamination insert. The delamination-resistance curves were similar for all three types of specimens.

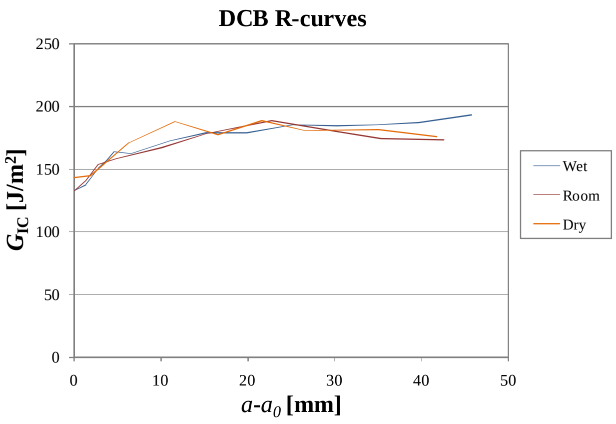


Fig. 9. DCB delamination-resistance curves

4.2 ENF test results

The critical energy release rate G_{IIC} insert values are illustrated in Fig. 10. The values showed mild increase with moisture.

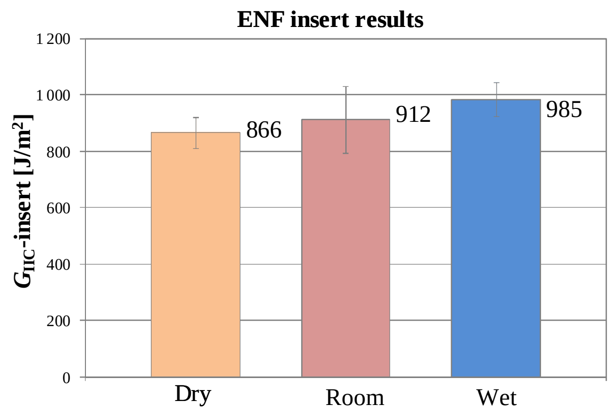


Fig. 10. ENF insert results

4.3 Discussion

The measured mode I insert values suggest that the increasing moisture content increases the interlaminar fracture toughness. The mild increase in the G_{IIC} insert values with increasing moisture content also support this conclusion. On the other hand mode I precrack values behaved differently when moisture content increased.

Considering the findings it can be concluded that the increase in the laminate moisture content yields to higher interlaminar fracture toughness values when the measurement is taken from the resin-rich insert tip. As the increase in ductility of epoxy is typically observed in wet conditions the increase in G_{IC} can be related directly to the increasing cleavage toughness of the resin. The insert value may therefore be considered as a resin dominant fracture energy value. Generally the insert value can be argued to be the most conservative and meaningful estimate of the G_{IC} because it is not influenced by fibre bridging. However, the study suggests that the insert value can be susceptible when analyzing the environmentally exposed specimens.

The precrack results were quite similar for each series when the scatter of the results is taken into account. No notable differences between the measured R-curves were found (Fig. 7). Typically these values are affected by the intensity of fibre bridging which is associated to the fibre/matrix interfacial bond strength. Therefore it can be concluded that the exposures used in this study did not affect noticeably on the fibre bridging of interfacial bond strength.

The G_{IIC} values increased slightly with moisture. The crack shearing fracture mechanism is different from the crack opening fracture mechanism. It is obvious that larger crack surface in the mode II test produces greater fracture energy compared to the mode I energy. Fibre bridging does not interact to the crack propagation in the mode II test. The G_{IIC} value consists of the energy which is associated to the matrix shear cracking and the energy which is associated to the fiber/matrix interface fracture. The combination of the energy consuming mechanisms yielded to only slight increase in the average G_{IIC} value. The resin-rich area at the insert tip does not have clear effect on the ENF results in contrast to the DCB results. There was considerable scatter in the test results causing some uncertainty.

5 Summary and conclusions

In this study the goal was to find out the moisture effects on the AS4/3501-6 carbon/epoxy laminate G_{IC} and G_{IIC} values. The study did not focus on the test method or data reduction schemes. Simple calculation methods were used in the study. In calculation of the ENF test results with the corrected beam theory method material stiffness data presented in Table 1 was used. The stiffness values were measured at the ambient humidity. The error generated by the material data in the data reduction of the wet and dry test series was found insignificant in view of the inherent scatter of the ENF test.

With three different exposure conditions used in the study only small effects of moisture on the G_{IC} precrack and propagation values were observed. G_{IC} insert values were affected by the resin-rich area at the insert tip. G_{IIC} values increased slightly with increasing moisture content. The study demonstrated that determination of interlaminar fracture toughness reliably with existing test methods is challenging even when one environmental exposure parameter is altered. Typically there are many other parameters (etc. lay-up, temperature,…) affecting the delamination fracture resistance of a composite laminate. More research is needed for the experimental characterization of interlaminar fracture toughness with different parameters affecting delamination.

References

[1] Fibre-reinforced plastic composites – Determination of mode I interlaminar fracture toughness, G_{IC}, for unidirectionally reinforced materials. *International Organisation for Standardization, 15024:2001(E)*, ISO, 2001, 24 p.

[2] Adams D. F., Carlsson L. A. and Pipes R. B. *Experimental characterization of advanced composite materials*, 3rd edition, CRC Press LLC, Boca Raton, 2003, 238 p.

[3] Wang Y. and Williams J.G. Corrections for mode II fracture toughness specimens of composite materials. *Composites Science and Technology 43*, 1992, p. 251-256

[4] Testing methods for interlaminar fracture toughness of carbon fibre reinforced plastics. *Japanese Standards Association, K 7086*, JSA, 1993, 35 p.

[5] Hintikka P. Determination of interlaminar fracture toughness of composite laminates. *Master's thesis, Helsinki University of Technology, Department of Applied Mechanics*, 2009, 122 p.

[6] Standard test method for mode I interlaminar fracture toughness of unidirectional fibre-reinforced polymer matrix composites. *American Society for Testing and Materials International, D 5528-01*, ASTM, 2001, 11 p.

Acknowledgements

The research described in this paper was carried out at Aalto University School of Science and Technology, Department of Applied Mechanics under a contract with the Finnish Air Force.

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