INTERLAMINAR FRACTURE BEHAVIOUR OF TEXTILE COMPOSITES WITH THERMOPLASTIC MATRICES

S. G. Ivanov[a], L. Gorbatikh[a*], S. V. Lomov[a]

[a] *Department of Materials Engineering, KU Leuven, Kasteelpark Arenberg 44, Leuven, Belgium*
**Larissa.Gorbatikh@mtm.kuleuven.be*

Keywords: textile composites, fracture toughness, thermoplastics, carbon nanotubes.

Abstract

The motivation for the work is to compare the interlaminar behaviour of textile composites with the same reinforcement and three different thermoplastic matrices: PPS, PEEK and PEEK with carbon nanotubes. The interlaminar fracture toughness results obtained from double cantilever beam (DCB) and end notch flexure (ENF) tests show are unexpected considering our previous observations from tensile tests on the same composites.

1. Introduction

The interlaminar behaviour of textile composites with three thermoplastic matrices is investigated in the crack opening and shear modes, using the Double Cantilever Beam (DCB) test and the End Notch Flexure (ENF) test, respectively. The carbon fabric (**CF**) T300 3K 5Harness satin is chosen as reinforcement. This fabric is used by TenCate Advanced Composites (TCAC) for commercial composite material with Poly Phenylene Sulfide (PPS) matrix. TCAC produced laminates with the following three matrices:

– **PPS**: Fortron 0214 [1];

– **PEEK**: Poly Ether Ether Ketone, Victrex 150P [2];

– **PEEK+CNTs**: PEEK Victrex150P reinforced with 0.5 wt% of non-functionalized ("naked") carbon nano-tubes (CNTs) from Nanocyl (NC7000). CNTs are introduced into PEEK matrix by Vamp Tech.

The influence of these matrices on the composite tensile properties was earlier investigated in tension tests in the direction of warp yarns (Table 1), and in the bias direction (Table 2).

Type of laminate	**Normalized strength [MPa]**		**Ultimate strain, %**	
	Average	**St.dev**	**Average**	**St.dev**
CF/PPS	736	64	1.34	0.08
CF/PEEK	777	88	1.37	0.13
CF/PEEK+CNTs	693	56	1.21	0.03

Table 1. Results of tension tests in the warp direction for the 6-ply laminates. The strength is normalized to the fibre volume fraction of 50%.

Type of laminate	Maximal stress [MPa]		Ultimate strain, %	
	Average	**St.dev**	**Average**	**St.dev**
CF/PPS	243	4	17.9	0.08
CF/PEEK	313	8	20.9	0.13
CF/PEEK+CNTs	251	9	19.4	0.03

Table 2. Results of tension tests in the bias direction for the 6-ply laminates.

According to the tensile test results in warp direction, the matrices can be ranked in the following order from higher to lower performance: PEEK, PPS, PEEK+CNTs. In bias direction this ranking gives: PEEK, PEEK+CNTs, PPS. Tensile properties in the bias direction are more sensitive to the matrix type.

In the current work, these composites are further characterized on the subject of their resistance to delaminations. How will laminates with these three matrices be ranked for the interlaminar open mode (mode I) and shear mode (mode II) fracture toughness?

2. Sample preparation

The laminates for fracture toughness tests are produced by TCAC. Temperature of the consolidation process is 325°C for the laminates with PPS matrix, and 385°C for the ones with PEEK matrices. The average thickness and hence, the fibre volume fraction V_f of the produced laminates, somewhat differ as shown in Table 3.

Type of laminate	Volume fraction V_f, %	
	Average	**St. dev**
CF/PPS	54.2	0.4
CF/PEEK	55.2	0.3
CF/PEEK+CNTs	50.2	0.3

Table 3. The average fibre volume fraction in produced laminates.

The samples for both test types are cut with a water-cooled diamond saw from 12-ply laminates with $[(0,90)_3,(90,0)_3]_s$ lay-up. Upilex PI film of 13 µm thickness is inserted at the two edges of a plate as a pre-crack in warp direction (Figure 1).

Figure 1. A 12-ply plate of CF/PPS with 13 µm film inserted from the two sides of the plate.

The nominal width of a sample is 20 mm, the length is around 200 mm or longer. Aluminium blocks 20×20×15 mm are fixed with epoxy glue to the DCB specimen's end, where the starter crack is located.

3. Fracture toughness, mode I. Results of DCB tests.

The DCB test is done following procedures described in ASTM standard [3] for unidirectional composite. There is no specific standard for laminates with other lay-ups, or for textile laminates. The Instron 4505 machine is used in displacement control mode with the constant displacement rate 3 mm/min. A Limess camera is mounted on the top of travelling setup to allow for observation of the crack propagation on the painted side of a specimen in the window of 1.5 cm in length.

The crack in DCB tests of textile reinforced laminate usually propagates in jumps. A crack jump corresponds to a sudden drop of the force value. This type of behaviour for CF/PPS, was described earlier in [4,5]. After each drop, a new crack length is registered by the camera with the help of markers on a specimen side. Then the crack length *a* keeps a constant value, while the force and displacement are growing till the next crack jump and the force drop occur. The force – displacement curves (two samples for each type of laminate) are shown in Figure 2.

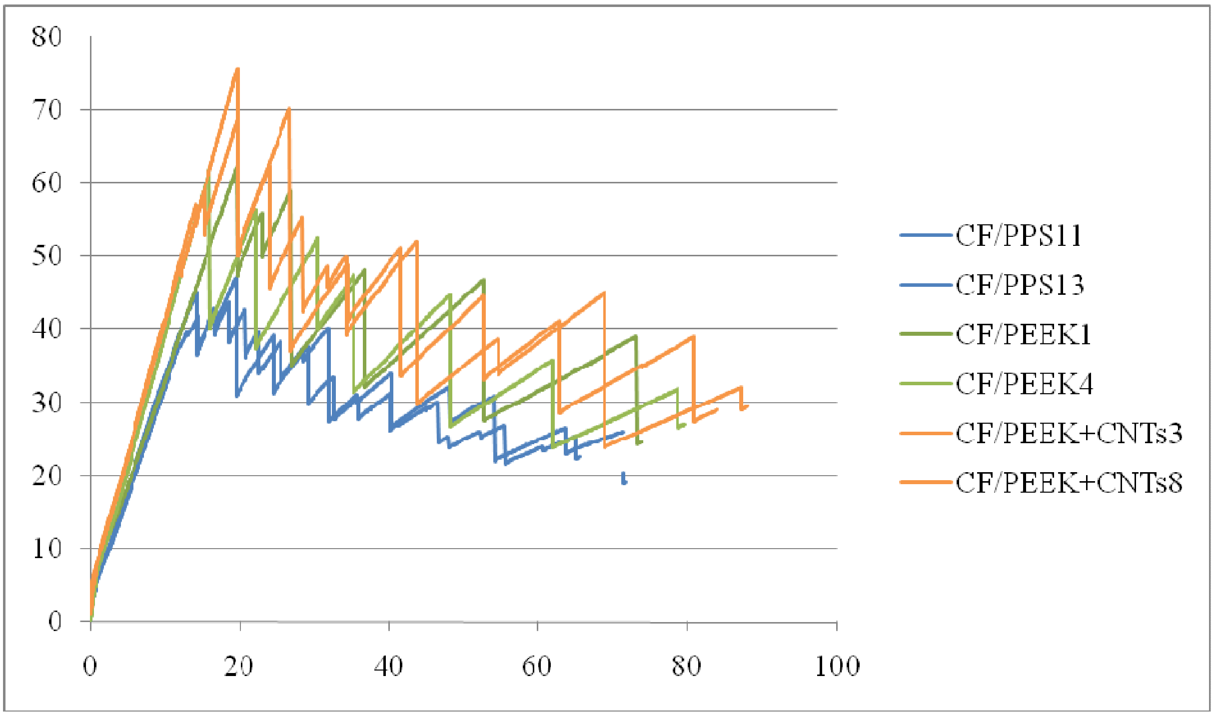


Figure 2. Force (N) – displacement (mm) curves in DCB tests.

The modified beam theory method is chosen for evaluation – the Mode I fracture toughness. The values of the strain energy release rate G_I are calculated **at peak points** of the force – displacement diagrams using the formula:

$$G_I = \frac{3 \cdot P \cdot \delta}{2 \cdot b \cdot (a + |\Delta|)} \cdot \frac{F}{N} \quad (1)$$

where P – force, δ – displacement, b – width of a sample, a – crack length,
F – correction factor for large displacement, N – correction factor to account for end block.
Δ is determined experimentally by generating a least squares plot of the cube root of compliance as a function of delamination length. The results of calculation are shown in Figure 3. Then the values from Figure 3 are averaged to calculate fracture toughness for the laminate, the results are shown in the Table 4.

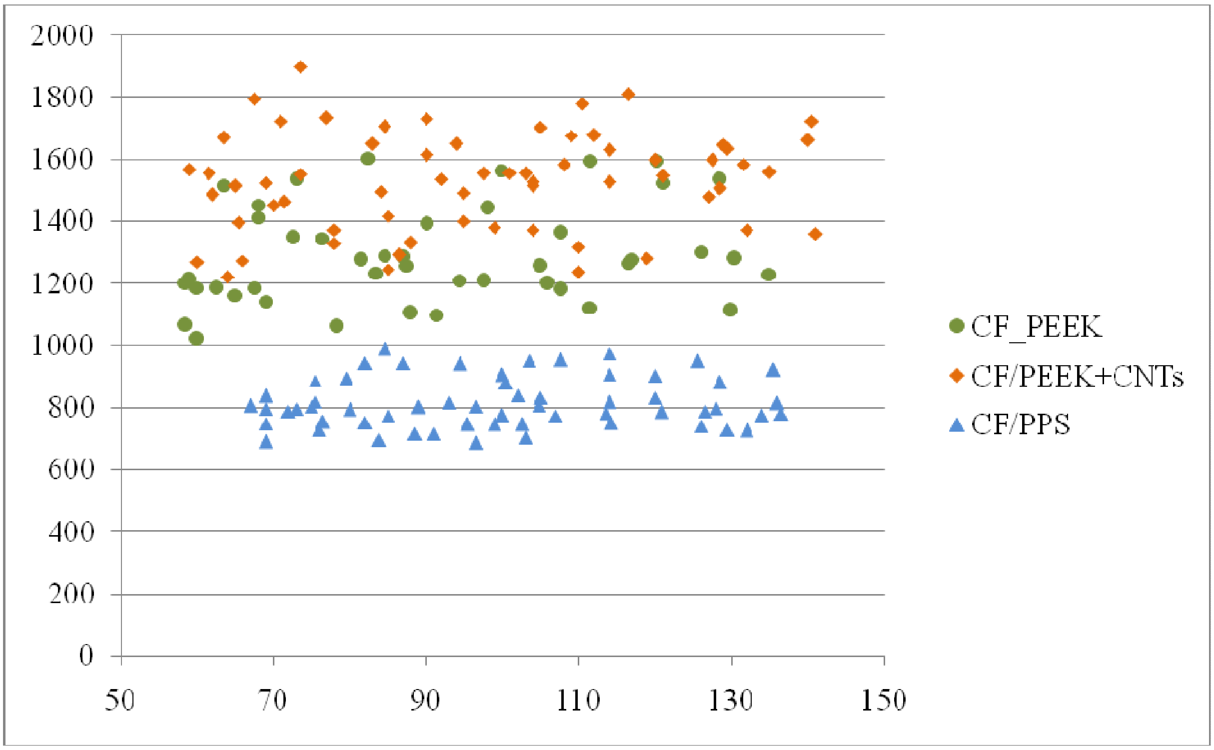


Figure 3. G_{Ic} values in the peak points (J/m^2) – delamination length a (mm) in DCB tests for three laminates

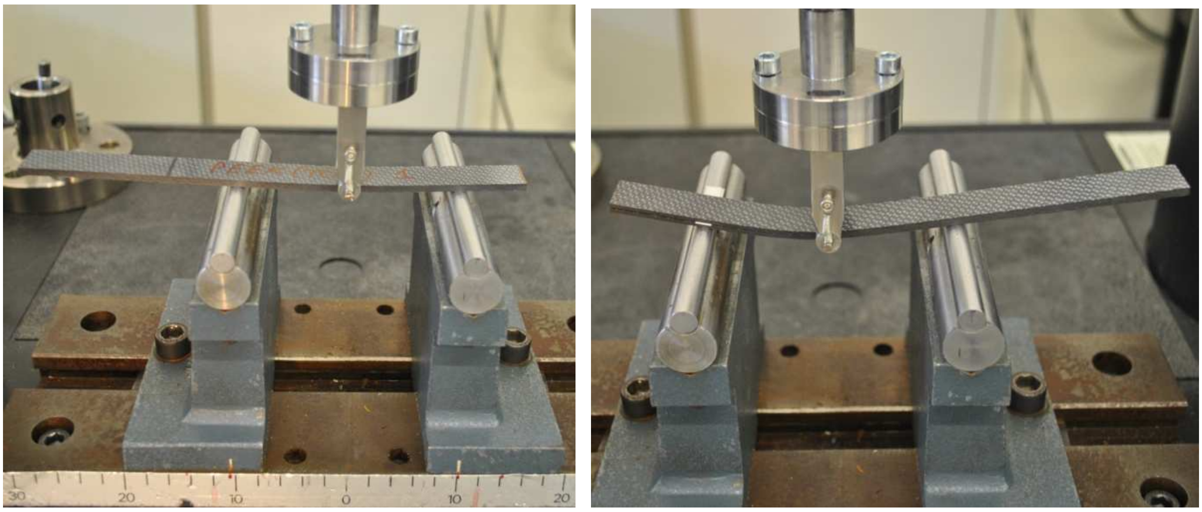
Type of laminate	G_{Ic}, kJ/m^2]		Number	Number
	Average	St.dev	of samples	of peaks
CF/PPS	**0.81**	0.08	6	59
CF/PEEK	**1.29**	0.16	7	44
CF/PEEK+CNTs	**1.53**	0.16	7	63

Table 4. Fracture toughness, mode I for textile laminates.

4. Fracture toughness, mode II. Results of ENF tests.

Interlaminar fracture toughness for mode II is measured usning the ENF test. The method is discussed in [5,6] and is used in [5] to study Mode II behaviour of 5-harness satin carbon fabric/PPS laminate from TCAC. The simple variant of this method is used in our study, without taking into account the influence of shear modulus and the change of the position of force application on the cylinder supports. But the method is modified as compared with [5] in another way. It requires determination of the compliance of the specimen without a crack before performing the ENF test. The used three-point flexure setup is shown in Figure 4. First, a laminate is tested in the shifted position, and the crack is placed outside the supports (Figure 4 a). The maximal load in this test corresponds to the strain ~0.3%. The compliance

C_0 of the laminate without any pre-crack is measured. Then the current compliance C is calculated in ENF test from force F – displacement δ graph: $C = \delta / F$.



a b

Figure 4. a – a preliminary test to measure the beam flexure compliance, b – ENF test.

For the main test, the laminate is shifted in the position with the initial pre-crack being a bit longer than 0.7 l (figure 4 b). For $a > 0.7l$ the crack propagation should be stable according to the beam theory. A 0.3 mm Teflon film is inserted in the pre-crack above the left support to reduce friction between the two surfaces of a crack in the ENF test. The loading nose and supports have cylinder surface with the radius 5 mm. The two values of a half of a span length l (the distance between the load line and a support) are used in this research: $l = 50$ mm for some tests and : $l = 70$ mm for the other tests.

Since the shear interlayer crack is difficult to be optically detected, the current equivalent crack length a_{eq} is estimated from (2):

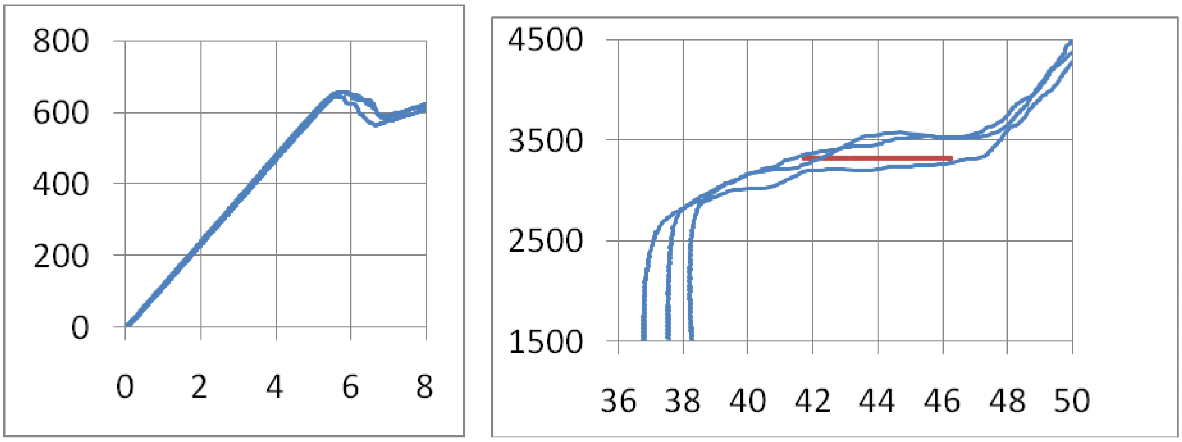
$$a_{eq} = l\left\{\frac{2}{3}\left(\frac{C}{C_0} - 1\right)\right\}^{\frac{1}{3}} \quad (2)$$

Then the value of strain energy release rate G_{II} can be calculated from (3):

$$G_{II} = \frac{9 \cdot a_{eq}^2 \cdot F^2 \cdot C_0}{4 \cdot w \cdot l^3} \quad (3)$$

The calculated G_{II} is plotted as a function of crack length a_{eq}. For the stable crack propagation this dependence should show a plateau, the value of this plateau is the value of the interlaminar fracture toughness G_{IIc} for crack propagation.

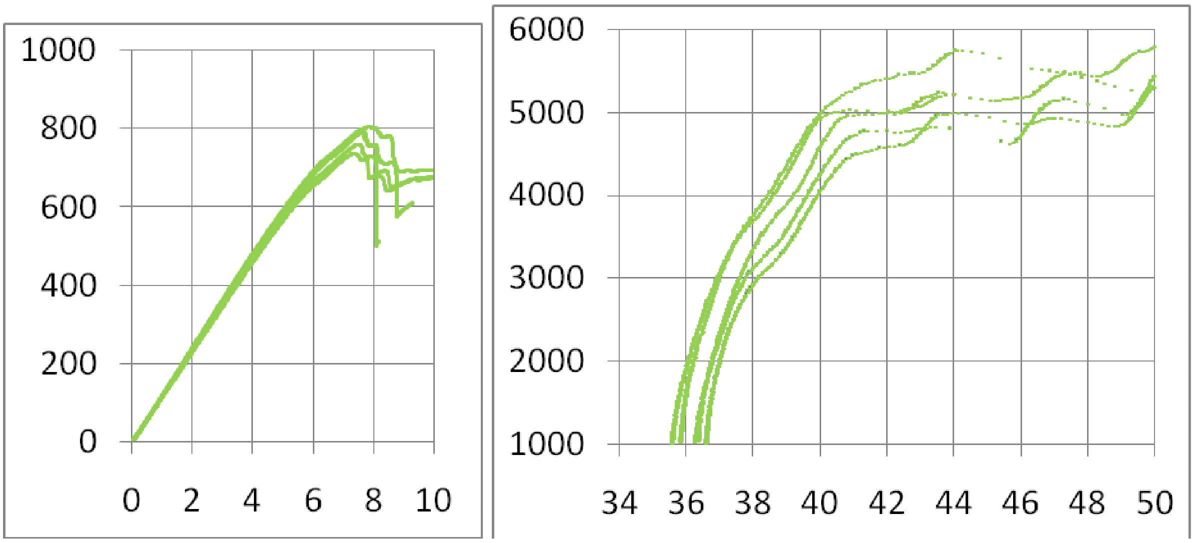
The force – displacement curves for 3 samples of the laminates with PPS matrix and the corresponding $G_{II} - a_{eq}$ curves are shown in Figure 5. The crack propagation is stable, as this was earlier investigated in [5]. The obtained value of $G_{IIc\ propagation}$ is quite close to the value for the same CF/PPS laminate in [5].



a b

Figure 5. ENF test for laminate with PPS matrix, span 100 mm. *a* – Force(N) – displacement (mm); *b* - G_{II} (J/m^2) – crack length a_{eq} (mm). Red line corresponds to $G_{IIcpropagation}$.

Comparing the results for CF/PEEK laminates (Figure below) with the results for CF/PPS laminates, we also observe the plateau with the higher level of fracture toughness for CF/PEEK laminate. For some samples crack propagation includes crack jumps.

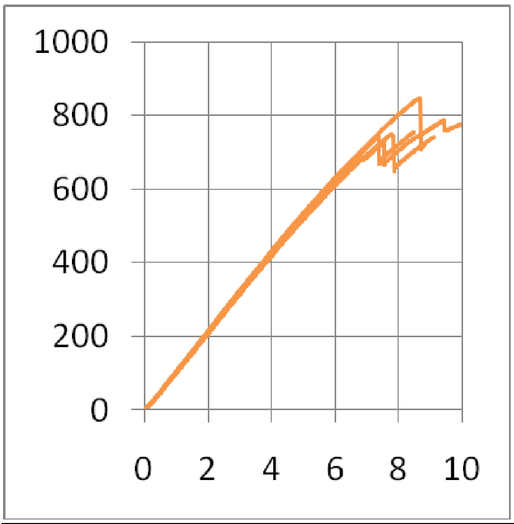


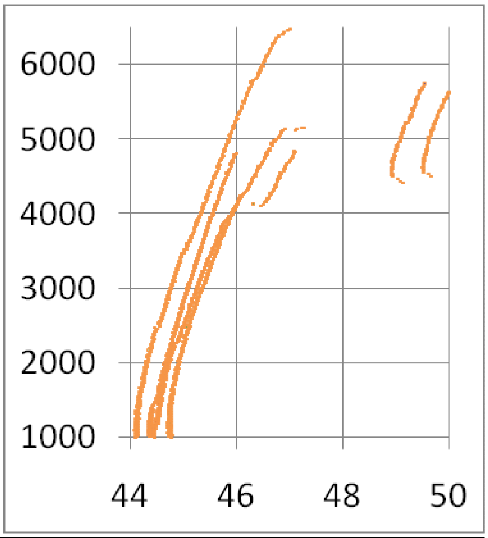
a b

Figure 6. ENF test for laminate with PEEK matrix, span 100 mm.
a – Force(N) – displacement (mm); *b* - G_{II} (J/m^2) – crack length a_{eq} (mm).

For the laminates with PEEK+CNTs matrix one can see (Figure 7) that after initial crack propagation for 2-3 mm the next crack propagation is unstable, the probable plateau is not reached. The $G_{IIc\ propagation}$ value is defined in this case as the maximal G_{II} value at which the stability is lost and crack jumps.

For the CF/PEEK and CF/PEEK+CNTs laminates the ENF tests have been performed with the span length 140mm as well. In Figure 6 and 7 G_{II} (J/m^2) – crack length a_{eq} (mm) curves are displayed. For the laminates with PEEK matrices strain energy release rate reaches the plateau – though for one of the presented samples the unstable big jump follows the stable behaviour. For laminates with PEEK+CNTs matrix no plateau is reached, the unstable behaviour starts earlier. The results of fracture toughness (mode II) evaluation are provided in Table 5.





a b

Figure 7. ENF test for laminate with PEEK+CNTs matrix, span 100 mm.
a – Force(N) – displacement (mm); *b* - G_{II} (J/m^2) – crack length a_{eq} (mm).

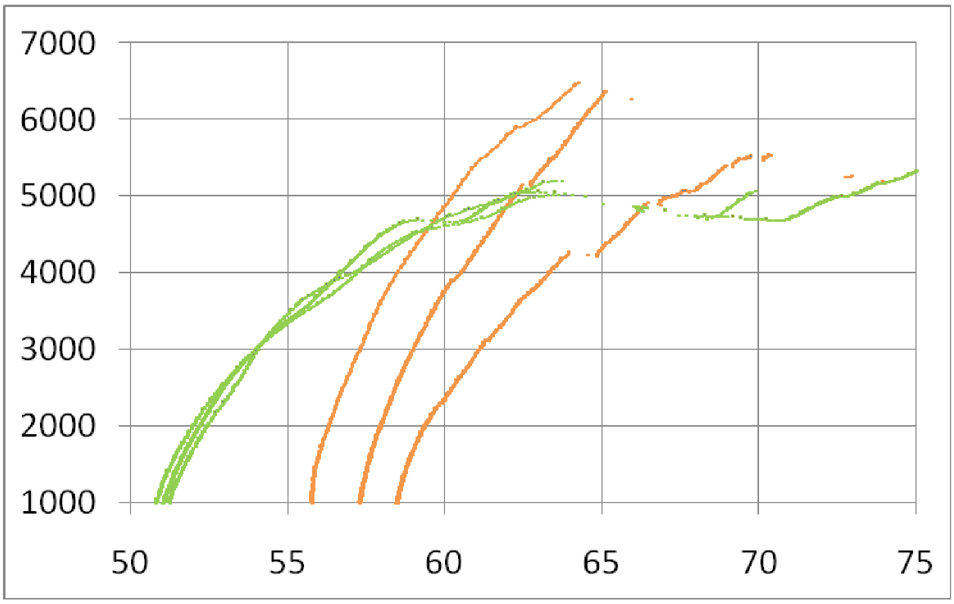


Figure 8. ENF tests for laminates with PEEK and PEEK+CNTs matrices, span 140 mm.
G_{II} (J/m^2) – crack length a_{eq} (mm).

Type of laminate	$G_{IIc\ propagation}$ [kJ/m^2]		Number of samples		
	Average	St.dev	Span 100 mm	Span 140 mm	Total number
CF/PPS	**3.32**	0.18	5	0	5
CF/PEEK	**5.14**	0.24	5	3	8
CF/PEEK+CNTs	**5.73**	0.68	6	3	9

Table 5. Fracture toughness, mode II for textile laminates.

5. Conclusion.

The character of the crack propagation in the open mode I is the same for the three laminates. The higher value of fracture toughness for CF/PEEK in comparison with CF/PPS was expected. But the bigger jumps of the crack propagation and the higher scatter of G_{Ic} in CF/PEEK in comparison with CF/PPS was unexpected. The order from higher to lower performance according to average G_{Ic} was also unexpected: **PEEK+CNTs, PEEK, PPS**.

Ranging the average values of $G_{IIc\ propagation}$, one obtains the same sequence of laminates for the shear mode II: **PEEK+CNTs, PEEK, PPS.** The average value for the CF/PEEK+CNTs textile laminate is more than 10% higher than the same value for the CF/PEEK laminate.
But CF/PPS and CF/PEEK laminates show the stable or close to the stable crack propagation if the crack length is more than 0.7*l*. The crack propagation for the CF/PEEK+CNTs laminate is always unstable. As a consequence of the crack propagation type, the scatter of $G_{IIc\ propagation}$ value for the CF/PEEK+CNTs laminate is approximately 3 times more than the scatter of the same value for the CF/PPS and CF/PEEK laminates.

Acknowledgements

This work is supported by the FP7 NMP program under the M-RECT project (246067) "Multiscale reinforcement of the semi-crystalline thermoplastic sheets and honeycombs". The authors thank the project partners Alan Wood (Victrex), Bruno Miani (Vamp Tech) for production of matrices and partners from TCAC: Renie Harbers, Gerjo Hultink, Sebastiaan Wijskamp for supplying laminates. The help of technicians Bart Pelgrims, Kris Van De Staey, Johan Vanhulst, Manuel Adams is gratefully acknowledged.

References

[1] Fortron® Polyphenylene sulphide (PPS). www.ticona.com
[2] Victrex® PEEK 150P. Victrex high performance polymers, 2012. www.victrex.com
[3] ASTM standard D 5528 – 01 Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites.
[4] V. Koissin, L.L. Warnet, R. Akkerman. Delamination in carbon-fibre composites improved with in situ grown nanofibres. *Engineering Fracture Mechanics* 2012; doi:10.1016/j.engfracmech.2012
[5] I. De Baere, S. Jacques, W. Van Paepegem, J. Degrieck. Study of the Mode I and Mode II interlaminar behavior of a carbon fabric reinforced thermoplastic. *Polymer Testing*, 31: 322-332, 2012.
doi:10.1016/j.polymertesting.2011.12.009
[6] A. Arrese, N. Carbajal, G. Vargas, F. Mujika. A new method for determining mode II R-curve by the End-Notched Flexure test. *Engineering Fracture Mechanics*, 77: 51-70, 2010. doi:10.1016/j.engfracmech.2009.09.008