INTERLAMINAR STRENGTH AND 3D REINFORCEMENT OF CARBON-POLYAMIDE 12 COMPOSITE MATERIAL

M. W. Tahir , S. Hallström , M. Åkermo

KTH Royal Institute of Technology, Department of Aeronautical and Vehicle Engineering, SE-10044 Stockholm Sweden

**Corresponding author: mwtahir@kth.se*

Keywords: Thermoplastic commingled composites, microscopy, interlaminar fracture toughness.

Abstract

Thermoplastic composite materials have some advantages over thermoset resin based composites, such as fast processing, recyclability, reparability etc. In the present study commingled Carbon/Polyamide 12 (PA12) is analyzed as dry yarn and after heating the matrix fibers above the melting temperature and applying ambient pressure. The results are presented as micrographs. The mode I interlaminar fracture toughness of Carbon PA12 composite materials is measured, with and without introducing through-thickness yarns, and it is found to be very high even without 3D reinforcement. The consolidation time is also found to have great effect on the measured fracture toughness.

1. Introduction

1.1. *Background*

Thermoplastic composite materials have advantages compared to thermoset composites in terms of higher toughness, lower processing time, reparability and ability for recycling, as well as health issues in manufacturing.

The processing of the thermoplastic composite material is carried out by first heating the fabric mat above the matrix melting temperature and then applying pressure, and finally cooling the material until the matrix solidifies while maintaining the pressure [1]. PA12 belongs to the Nylon family and it bonds very well to itself, which may provide good delamination properties.

1.2. *Scope*

The aim of this study is threefold:

- to determine an effective manufacturing process based on understanding of the wetting process,
- to study the influence of consolidation on the interlaminar delamination of Carbon PA12 composites,
- to investigate the influence of 3D reinforcement on the interlaminar properties.

To the knowledge of the authors delamination properties of Carbon PA12 material has not been studied thoroughly before. The study is further aimed at exploring whether 3D reinforcement would be favourable for this material or not.

2. Experimental

2.1. Materials

The material used in the present study is Carbon PA12, Nm 3.2 (equivalent to 3K carbon yarn), with a 55% fibre volume fraction of carbon with an average length of 80 mm, the commingled material is provided by Schappe Techniques.

2.2. Sample preparation

Layers of the fabric are cut and stacked on each other making sure that all layers are aligned properly. The material is then put into a conventional oven heated to 220°C with a temperature sensor placed in the middle of stack. In order to generate the initial crack, a strip of a 0.025 mm thick release film is placed along one edge between the middle layers. When the temperature of the lamina reaches 220°C, the material is moved to a hot press with top and lower platens preheated to 220°C, and a pressure of 20 bar is applied. The material is kept under constant pressure and temperature for 3 minutes, then the cooling cycle is started with cooling rate of 15°C/min, with maintained pressure. The consolidation process is illustrated in Fig. 1. After cooling, the laminate is taken out and double cantilever beam (DCB) specimens are prepared.

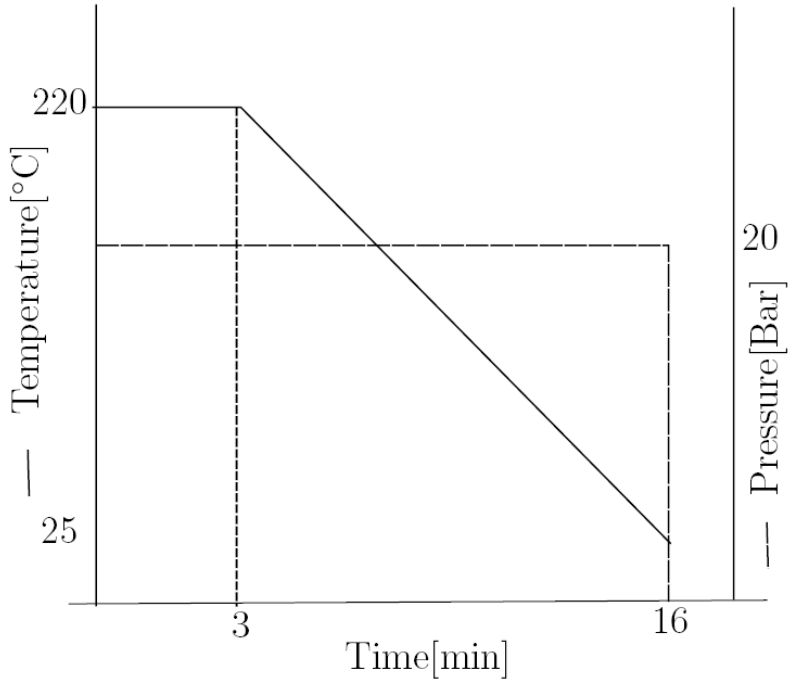


Figure 1. Consolidation process for Carbon/PA12 composite material.

2.3. Microscopy

Yarns are heated above the melting temperature of the PA12. Single dry as well as melted yarns are then immersed in a mould filled with liquid epoxy resin so that they remain straight while the resin hardened. After curing of the epoxy, the specimens are polished and examined in a microscope.

In order to study the consolidation results microscopy is also performed on the laminates from the press.

2.4. Delamination Tests

ASTM standard D 5528 is followed for the DCB tests. The standard provides guidelines for determination of the mode I crack energy release rate. The load is introduced either via piano hinges or pins through bonded end-blocks. Fig. 2 schematically illustrates a DCB specimen with piano hinges. Different samples are prepared as shown in Table 1, for all the specimens except one *(EB2)* the processing conditions are as described before. For the specimen *EB2*, the consolidation time is doubled to 6 minutes.

Name	**No. of Plies**	**Thickness *t* [mm]**	**Width *b* [mm]**	**Initial crack length *a0* [mm]**
PH1	12	2.93	26.5	46.5
PH2	16	3.75	27.9	52
PH3	20	4.5	27.0	45
PH4	32	7.1	24.8	109
EB1	32	7.1	25.1	25.2
EB2	32	7.05	25.6	25.7
PH – piano hinge EB – end block with pins				

Table1 1. Various specimens used for DCB tests.

The initial crack tip is marked and measured from the load line, with a typing correction fluid applied on the sides of the specimens to help visualising the crack front. The free end of the DCB test specimens is initially supported, as shown schematically in Fig. 3. The crack growth is monitored and measured using a sliding microscope travelling on a scale parallel with the crack path. During the loading, the specimens move upwards, making the free end leaving their end support. However the free ends remain horizontal throughout the tests and the support is not needed during loading.

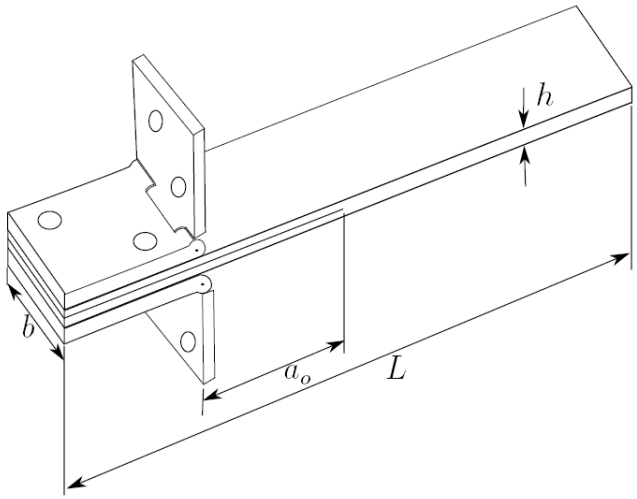


Figure 2. DCB test specimen with piano hinges.

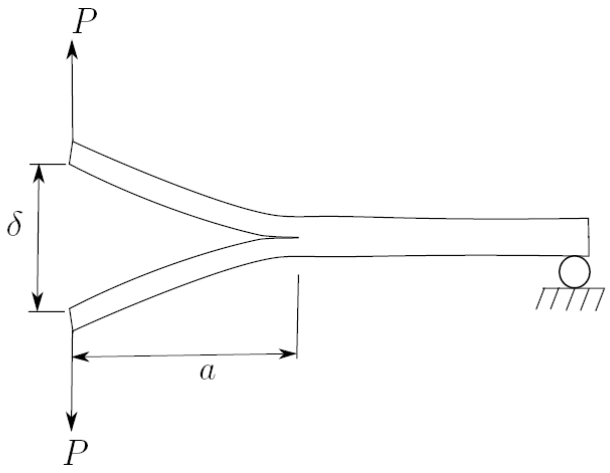


Figure 3. Loading of the DCB, schematic diagram.

The expression for G_I of the perfectly built in beam is:

$$G_I = \frac{3P\delta}{2ba} \tag{1}$$

where, P is the applied load, δ is the vertical displacement and a is the delamination length as shown in Fig. 3, and b is the width of the specimen.
Practically, some rotation may occur at the crack front making the expression for G_I in Eq. (1) overestimating G_I slightly. To cope this problem a correction suggested in the ASTM standard 5528 is as follow:

$$G_I = \frac{3P\delta}{2b(a+\Delta)} \tag{2}$$

where, Δ is the x-intercept of the least square plot of the cube root of compliance ($C = \delta/P$) versus the delamination length.

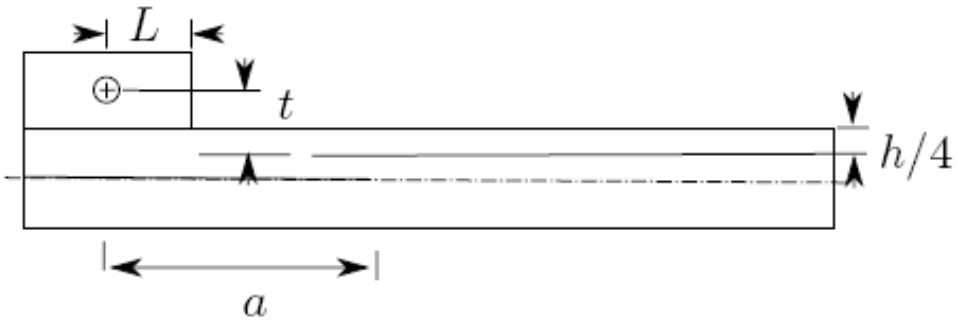


Figure 4. DCB specimen with end blocks.

While using the end blocks correction factors F and N are suggested in the ASTM standard as follows:

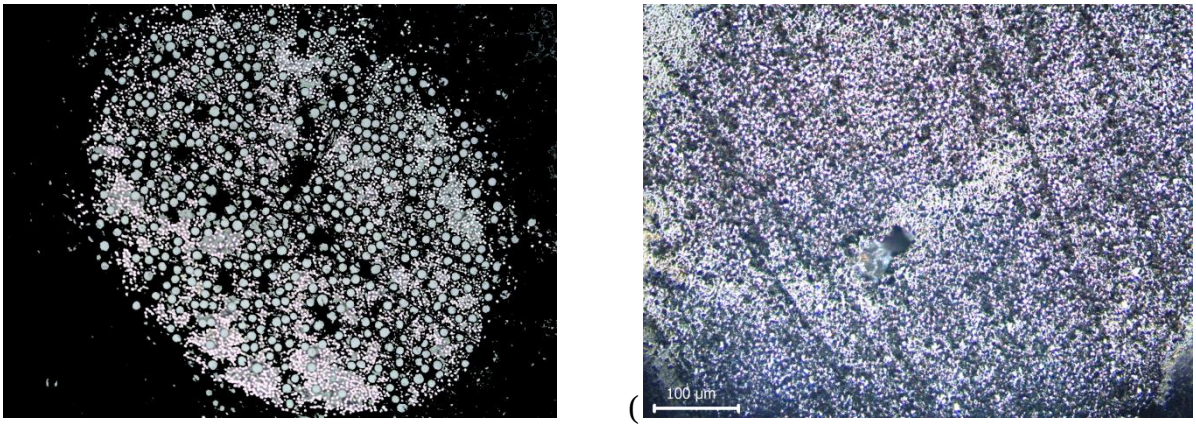
$$F = 1 - \frac{10}{3}\left(\frac{\delta}{a}\right)^2 - \frac{3}{2}\left(\frac{\delta t}{a^2}\right) \quad (3)$$

$$N = 1 - \left(\frac{L}{a}\right)^3 - \frac{9}{8}\left\{1 - \left(\frac{L}{a}\right)^2\right\}\frac{\delta t}{a^2} - \frac{9}{35}\left(\frac{\delta}{a}\right)^2 \quad (3)$$

where t, a and L are as shown in figure 4. The compliance C, is then divided by N, and G_I is multiplied by F/N, for details the reader is referred to the ASTM standard D 5528.

3. Results and Discussion

Micrographs of the virgin single yarn show that there is homogeneous mingling of carbon and *PA12* fibres in the yarn. In Figs. 5a and 5b photographs of a dry yarn and a yarn heated to matrix melting temperature at atmospheric pressure are shown, respectively. In figure (5a) *PA12* fibres are visible as larger dots (with a diameter of about 20μm), whereas the carbon fibres are visible at smaller dots (with a diameter of about 7 μm).



(a) (b)

Figure 5. Micrographs of virgin yarn (a) and yarn with melted matrix (b).

A micrograph of a six layer laminate is shown in Fig. 6, showing reasonably good consolidation. It is observed during the DCB tests that the Carbon *PA12* material is very tough and not prone to delaminate at all. In fact, it is truly challenging to propagate delamination cracks in the material within the frames set by the DCB test standard. The first three samples *PH1*, *PH2* and *PH3* failed in bending before the crack started to propagate. Then for specimen *PH4* with 32 plies, the crack first grew rapidly and then arrested after about 16 mm propagation. Then it failed in bending and the test was stopped. G_I computed using Eq. (1) for the available data points is shown in Table 2.

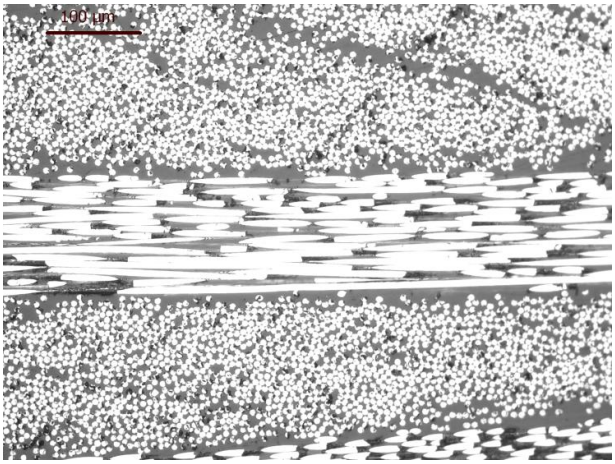


Figure 6. Micrograph of 6 layers of Carbon PA12 laminate.

For the specimen *EB1* the initial crack length was reduced to 25.2 mm avoid bending failure before crack propagation. For this specimen the crack grew in 6 increments until the specimen failed in bending at a total crack length of 87 mm. The G_I results from the specimen are presented in Fig. 7.

Specimen	***PH4***
Load, *P* [N]	153.88
Delamination length, *a* [mm]	125
Width, *b*, [mm]	24.8
Vertical displacement δ, [mm]	40.94
Interlaminar fracture toughness, G_I [J/m^2]	3048.31

Table 2. *GI*, value for specimen *PH4*, with only one valid data point.

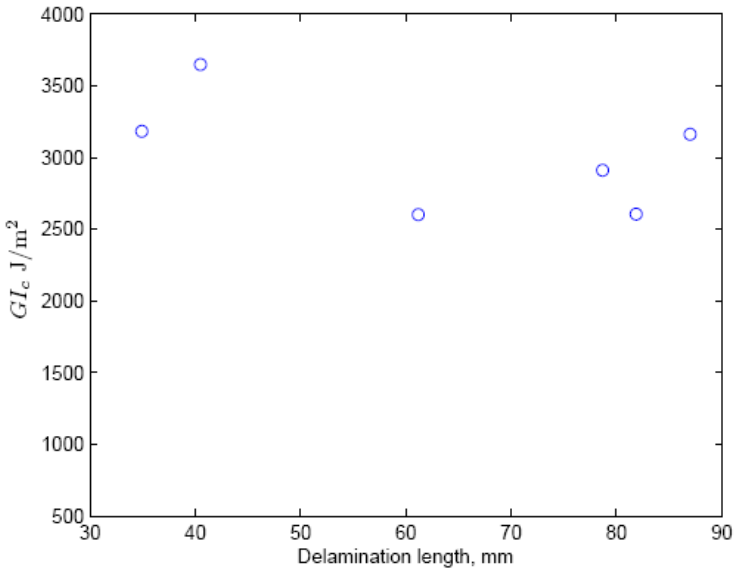


Figure 7. G_I versus delamination length for specimen *EB1*, after end-block compensation.

The crack propagated straight between layers in the specimen. For comparison, round robin tests [2] reported G_I values for AS4/PEEK, a notably tough thermoplastic material, in the range of 1300-2000 J/m^2. The present values exceeds all values reported for AS4/PEEK and are about 5 times higher than what is typical for carbon/epoxy laminates. From these preliminary results it appears like Carbon PA12 has very high interlaminar fracture toughness. Delamination might thus not be a critical issue for this material, provided that it is well consolidated.

The last specimen in Table 1, *EB2*, is different from the other specimens in that the consolidation time was changed from 3 to 6 minutes. In this specimen the crack grew properly and no bending failure occurred. The specimen provided 11 valid crack growth increments used in the evaluation.

G_I values versus delamination length from specimen *EB2* are shown in Fig. 8. Clearly, the G_I values of this sample are much lower than for the *EB1* specimen. It is assumed that the increased consolidation time somehow affected the delamination strength but further investigation is needed to determine what caused the difference. Some matrix accumulation is seen on the edges of the *EB2* specimen, indicating that resin has started to seep out from the composite material. The effect of the consolidation time on the properties of the laminate is a key parameter for efficient processing. The drop of toughness for higher processing time suggests that there is an optimal process time somewhere in the range 0-6 minutes, for the given material and circumstances. The aim of the continued work is to investigate this further, focussing on how much the processing time can be decreased without loosing material performance. In addition the aim is to try to compensate for the diminished fracture toughness by introducing through-thickness yarns in the reinforcement.

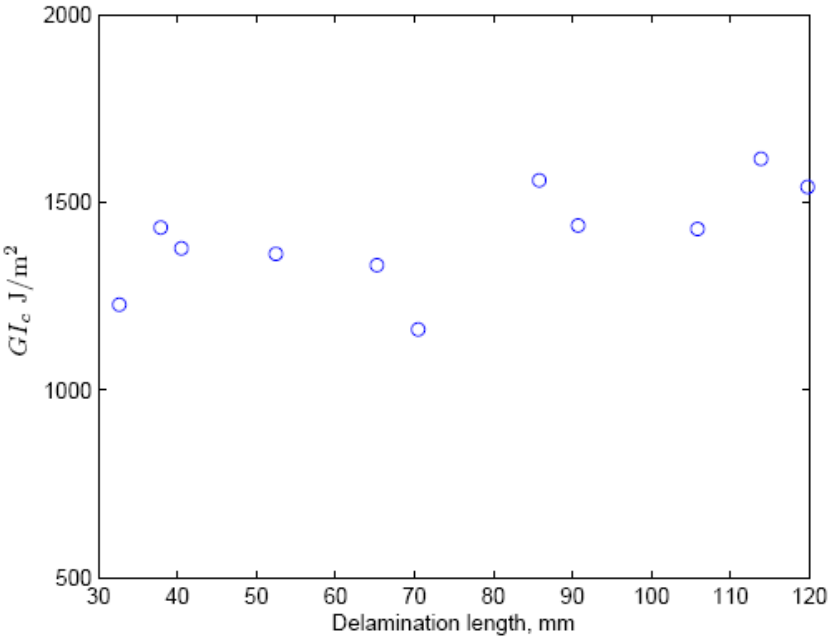


Figure 8. G_I versus delamination length for specimen *EB2*, after end-block compensation.

4. Conclusion

Microscopy images show that the matrix material is evenly distributed in the Carbon PA12 material, both in dry yarns and after the yarns have been heated above the matrix melting temperature. It appears like capillary forces play an important role in the resin distribution and the wetting of the reinforcement after melting. However, the application of pressure is necessary for complete consolidation of the laminate.

DCB test results indicate that the material has very high interlaminar fracture toughness as compared to results for carbon/epoxy and even carbon/PEEK composites. Moreover, it can be stated that the consolidation time appears to play an important role for the fracture toughness of the material. However, further investigation is needed to make solid and statistically sound conclusions.

REFERENCES

[1] N. Bernet, V. Michaud, P. E. Bourban, and J. A. E. Manson, An impregnation model for the consolidation of thermoplastic composites made from commingled yarns, *Journal of Composite Materials,* **33(8):751-772, 1999.**

[2] O'Brien T. K. and Martin R. H., Round robin testing for mode I interlaminar fracture toughness of composite materials, *Journal of Composites Technology and Research,* **15(4):269-281, 1993.**