INFLUENCE OF THE CONSOLIDATING PRESSURE OF THERMOPLASTIC PREPREGS ON THE MODE I INTERLAMINAR FRACTURE TOUGHNESS

Avril C. [1 2], Reck B. [1], Perreux D. [2], and Thiebaud F. [2]

[1] Dept. of Electromagnetic Acceleration, French-German Research Institute of Saint-Louis, 68301 Saint-Louis Cedex, France
[2] Applied Mechanics Laboratory Raymond Chaléat – FEMTO-ST, University of Franche-Comté, 25000 Besancon

ABSTRACT

In the framework of the mechanical behavior optimization of the PEGASUS railgun supporting structure, the influence of the consolidating pressure of S2-Glass/PEEK and E-Glass/PEEK prepregs on the mode I interlaminar fracture toughness G_{Ic} is investigated. Three different data reduction methods are presented and used to determine the initiation and propagation values of G_{Ic}. Lower consolidating pressures, i.e. higher resin contents, lead to a better delamination resistance even if branching phenomena observed for the highest pressures slow down significantly the crack growth. The delamination of E-Glass reinforced specimens has proved more unstable but the G_{Ic} mean value obtained was much higher than for the S2-Glass reinforced specimens.

1. INTRODUCTION

The PEGASUS electromagnetic railgun facility at ISL (figure 1-a) is designed to accelerate projectiles with masses of 1 kg to velocities of about 2500m/s. In order to achieve such a performance the electrical shot-parameters and the mechanical resistance of the projectile have to be optimized. The projectile is composed of a payload, a payload supporting structure and electric brush armatures (figure 1-b). The 40 mm thick supporting structure should be an electrical insulator and also have a low mass and a high mechanical strength. Therefore, a polymer resin impregnated glass fabric laminate is used. X-Ray flash radiographs of the projectile during the acceleration and outside of the launching tube allow to follow the evolution of the structure. The experiments showed that the most common damage mode is delamination in mode I. A loss of layers causes a less efficient support of the armature leading to a deterioration of the electrical contact between the brushes and the rails. The latter can lead to the formation of electric arcs (plasma) which itself provokes the destruction of both the rails and the projectile.

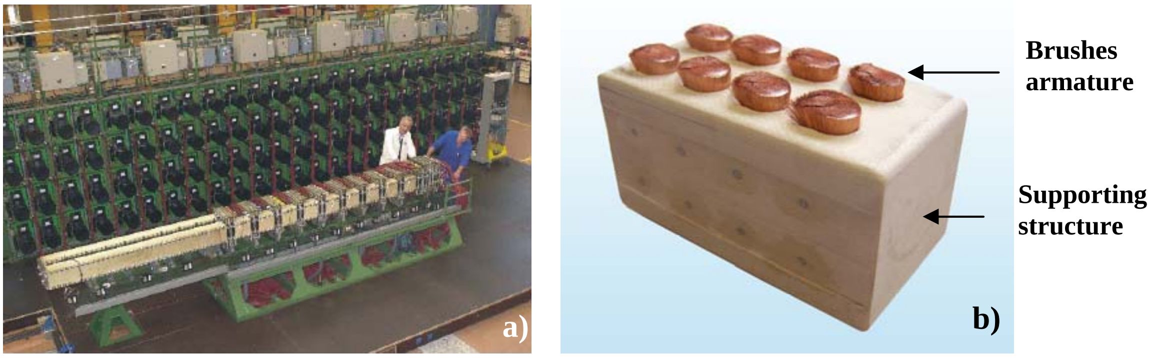

Figure 1: PEGASUS facility at ISL (a) and PEGASUS projectile without payload (b)

Up to now epoxy resin impregnated E-Glass fabric were mainly used for the fabrication of the laminate. In order to improve the interlaminar fracture behaviour of the supporting structure the epoxy resin has been replaced with a more ductile thermoplastic resin: PEEK. The first laminates were based on preimpregnated S2-Glass woven fabrics. The 196 prepreg layers were consolidated by a supplier with a moulding temperature of 390°C and a pressure of 10 bars for 30 min. These laminates have shown a better resistance to delamination than previous Glass/Epoxy laminates and allowed to reach velocities of about 2500 m/s [1]. However delaminations were still observed and the limits of the material seemed to be reached. In the perspective of having a better control on the properties of the supporting structure, the most recent laminates have been self-manufactured. As a semi-crystalline polymer, PEEK offers the possibility to influence the mechanical characteristics of the polymerized composite by changing the processing parameters: molding temperature, cooling rate or annealing conditions have significant effects on the matrix degree of crystallinity and thus on its mechanical behavior [2] [3] [4]. Compared to the industrial process, two parameters have been mainly changed for the manufacture of our first laminates: an increased consolidating pressure of 30 bars and a slower cooling rate of about 1.5°C/min. The comparison between the shot results obtained with industrial and self-manufactured laminates is unequivocal: whereas the industrial supporting structures accelerated with a total electrical energy of 2.57 MJ are delaminated (figure 2a), the limits of the self-manufactured projectiles haven't been yet reached. The most recent shot involved a total electrical energy of 3.32 MJ and the composite structure remained intact when it left the launching tube (figure 2b).

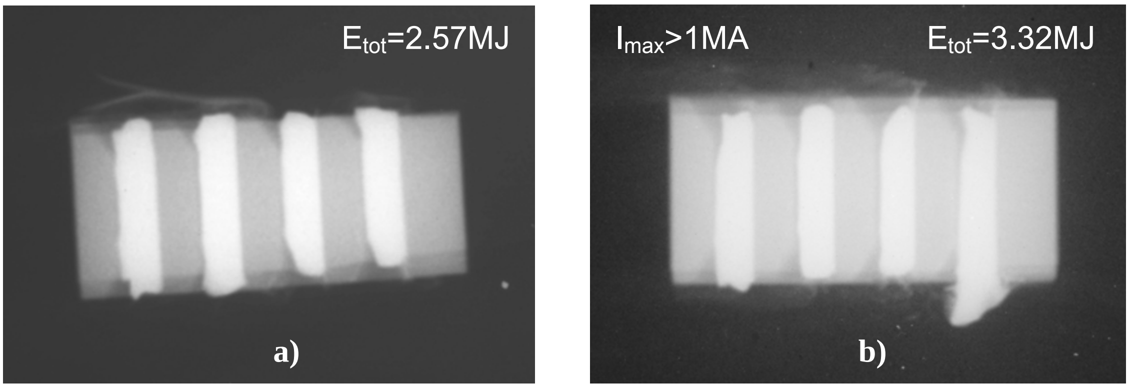

Figure 2: X-Ray flash radiographs of the delaminated industrial projectile (a) and intact self-manufactured projectile (b) after the projectile left the launching tube

In order to explain this improvement of performance, two points related to the manufacturing process are currently explored. The first one deals with the effect of the cooling rate on the laminate mechanical resistance. This paper concerns the latter one, namely the influence of the consolidating pressure on the mode I interlaminar fracture toughness of S2-Glass fabric-reinforced PEEK matrix composites. The consolidating pressure is directly related to the polymer resin content of the laminate. What this study actually investigates is how the amount of resin between the different layers acts on the interlaminar toughness. It could also lead to important weight-savings for the supporting structure: for a given laminate thickness, a higher molding pressure means a higher number of layers. Considering the superior density of glass fibres compared to PEEK, optimizing the pressure could allow to optimize the weight of the projectile and as a consequence its velocity. The standard test method using double cantilever beam (DCB) specimens has been followed [5] [6].

2. PREPARATION OF THE DCB SPECIMENS AND TESTS PROTOCOL

Two materials were investigated: PEEK preimpregnated E and S2-glass balanced woven fabrics. These two materials were chosen owing to their similar characteristics (table 1). The present study concerns more specifically the S2-glass prepreg whose tensile resistance is of particular interest for the railgun application, but the E-glass prepreg was expected to give relevant information about the role of the fibre/matrix cohesion in the delamination process.

Style	*Weave*	*Construction Warp/Weft (yarn/cm)*	*Dry Fabric Areal Weight (g/m^2)*	*Prepreg Areal Weight (g/m^2)*	*Polymer Content in Volume (%)*
E Glass 7581-P17	8H Satin	22.9*22.1	302	456	50
S2 Glass 6781-P17	8H Satin	22.8*21.3	300	460	50

Table 1: Characteristics of the two investigated glass fabric reinforced PEEK prepregs

The standard test method for evaluating the mode I interlaminar fracture toughness (G_{Ic}) of fiber-reinforced polymer matrix composites consists in applying a load perpendicular to the delamination plan to a rectangular laminated composite specimen containing a nonadhesive insert on the midplane aimed at initiating the delamination (figure 3).

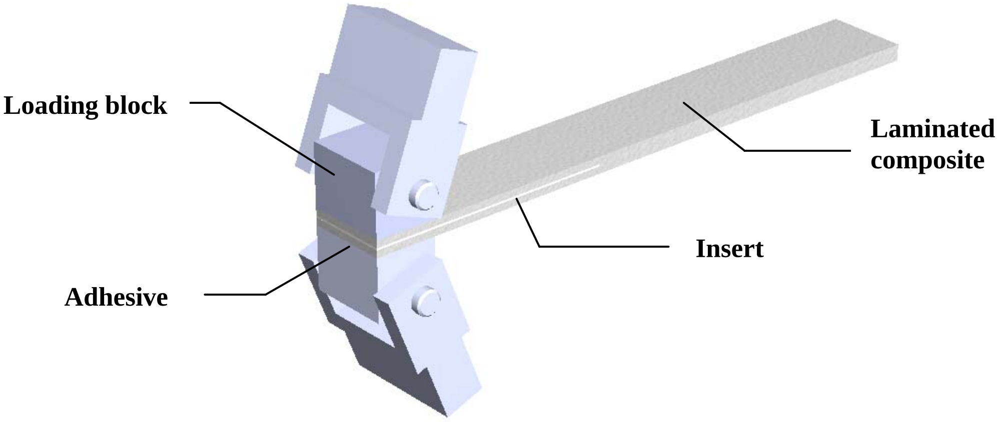

Figure 3: Double Cantilever Beam Specimen

The specimens were built by stacking 26 layers of prepregs in a 172 mm long and 100 mm wide steel mold. A very thin 70 mm long PTFE film was used as a delamination starter. The same pressing system as for the realization of the PEGASUS supporting structures was used: a standard oven adapted to an Instron ±100 kN tensile machine. A force of -10 kN (equivalent to 6 bars in regard of the mold dimensions) was applied until a temperature of 380°C was reached. The compression force was then increased depending on the intended pressure and hold for 30min. The cooling was simply achieved by opening the oven door in order to obtain a slow cooling rate. Four different consolidating pressures were applied: 6, 10, 20 and 30 bars. The thickness of the so-obtained plates logically decreased with increasing molding pressure (figure 4). The consolidated ply thickness was of about 0.219 mm for 6 bars, 0.204 mm for 10 bars, 0.190 mm for 20 bars and 0.184 mm for 30 bars. Each plate was then trimmed and three 152 mm long and 20 mm wide DCB specimens per plate were milled by means of carbide tools. Bonding of the loading blocks was performed by using a two-component epoxy adhesive (Loctite 9466). Specimen edges were marked at regular intervals to facilitate the monitoring of the delamination propagation. Each test was divided into two steps: a first load aiming at propagating the delamination from the insert on a length of about 5 mm, followed by a second load propagating the delamination from the pre-crack until a delamination length of about 50 mm was reached (figure 5). The load and the opening displacement were systematically recorded and stored in the perspective of the data post-processing.

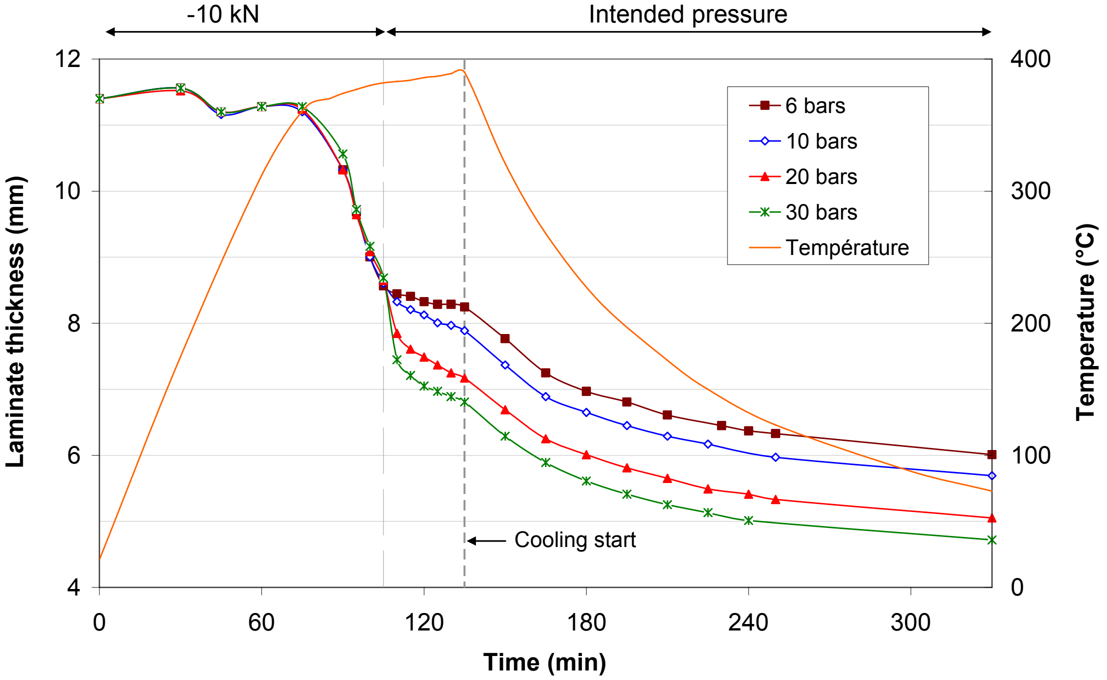

Figure 4: Evolution of the laminates thickness in regard of the temperature and molding pressure during the manufacturing process.

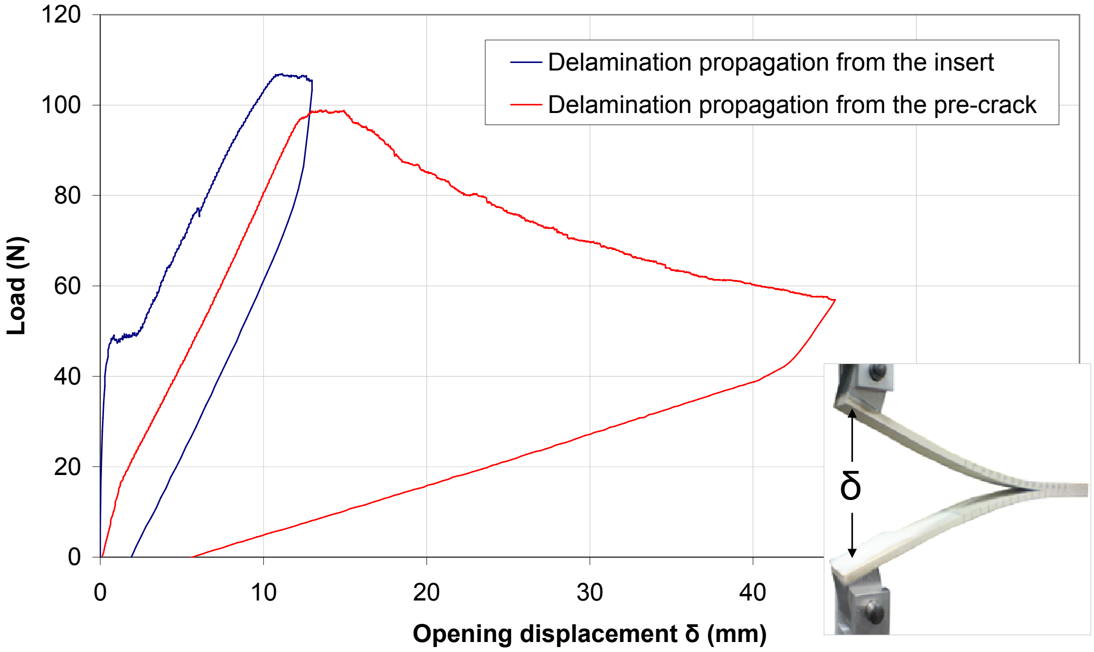

Figure 5: Typical load versus opening displacement curve obtained for DCB tests

3. THEORITICAL BACKGROUND

Three data reduction methods have been employed to calculate the mode I interlaminar fracture toughness G_{Ic} of the glass/PEEK specimens: the simple beam theory (SBT), the modified beam theory (MBT) and the modified compliance calibration method (MCC). All these methods stem from the Irwin-Kies equation:

$$G_{Ic} = \frac{P^2}{2b}\frac{dC}{da}$$

where $C = \delta/P$ is the compliance, a the delamination length, P the applied load and b the specimen width. The three analysis schemes differ in the expression of the compliance C. The simplest G_{Ic} expression is given by the SBT:

$$G_{Ic} = \frac{3P\delta}{2ba}$$

For the calculation of the beam deflection, the SBT takes into consideration the contribution of shear and bending but ignores the beam rotation. The MBT introduces a factor Δ to correct this problem by assuming that the real delamination length is slightly longer than predicted by the SBT. Two additional factors are introduced to take into account the large displacements (δ/a>0.4) and the loading blocks stiffness (respectively F and N):

$$G_{Ic} = \frac{3P\delta}{2b(a+|\Delta|)} \times \frac{F}{N}$$

where $F = 1 - \frac{3}{10}\left(\frac{\delta}{a}\right)^2 - \frac{2}{3}\left(\frac{\delta \cdot l_1}{a^2}\right)$ and $N = 1 - \left(\frac{l_2}{a}\right)^3 - \frac{9}{8}\left[1 - \left(\frac{l_2}{a}\right)^2\right]\frac{\delta \cdot l_1}{a^2} - \frac{9}{35}\left(\frac{\delta}{a}\right)^2$

l_1 is the distance between the loading axe centre and the midplane of the specimen
l_2 is the distance between the loading axe centre and the edge of the loading block

The correcting factor Δ is determined experimentally by plotting the cube root of the ratio C/N as a function of delamination length a. The intercept of the curve with the X-axis gives the Δ value. The MCC method is based on a normalization of the delamination length by the specimen thickness e:

$$G_{Ic} = \frac{3m}{2e} \times \left(\frac{P}{b}\right)^2 \times \left(\frac{bC}{N}\right)^{2/3} \times F$$

where m is the slope of the plot of the ratio $(bC/N)^{1/3}$ as a function of $a/2h$.

The technical standard recommends to calculate three initiation values of GIc (figure 6). These values are determined using the load and opening displacement measured at the point of deviation from linearity in the load-displacement curve (NL), at the point at which delamination becomes visible under a microscope (VIS), and at the point at which either the compliance has increased by 5% or the maximum load value has been reached (5%/MAX). In our case, no microscope has been employed and in order to ensure the results accuracy, only the NL and 5%/MAX GIc values have been determined. The propagation values of GIc (PROP) are calculated using the load and opening displacement corresponding to the delamination lengths monitored during the tests thanks to the marks on the edge of the specimens. For each specimen, about ten propagation values of GIc were recorded. The NL and 5%/MAX initiation values of GIc weren't included in the linear regression for the calculation of the parameter Δ and m. Both initiation and propagation GIc values presented in this paper are determined from the pre-crack.

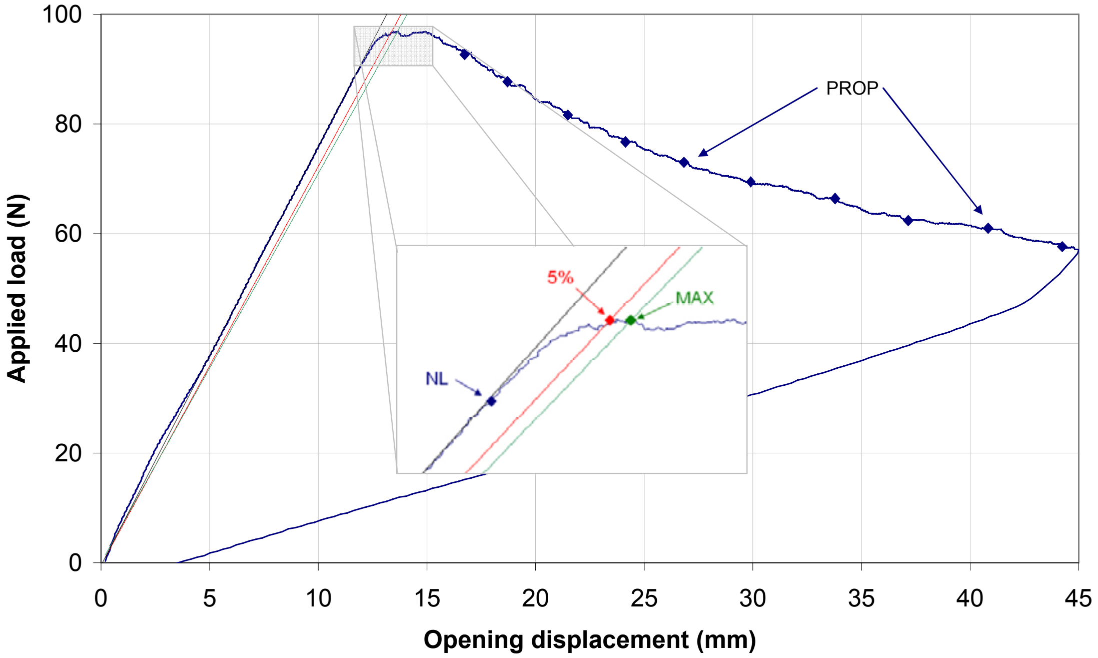

Figure 6: Definition of the initiation (NL and 5%/MAX) and propagation values of G_{Ic}

The figure 7 shows an example of a typical delamination resistance curve (R-curve). The general shape of this curve, obtained for a specimen consolidated with a 6 bars maximal consolidating pressure, is very representative of the majority of the results.

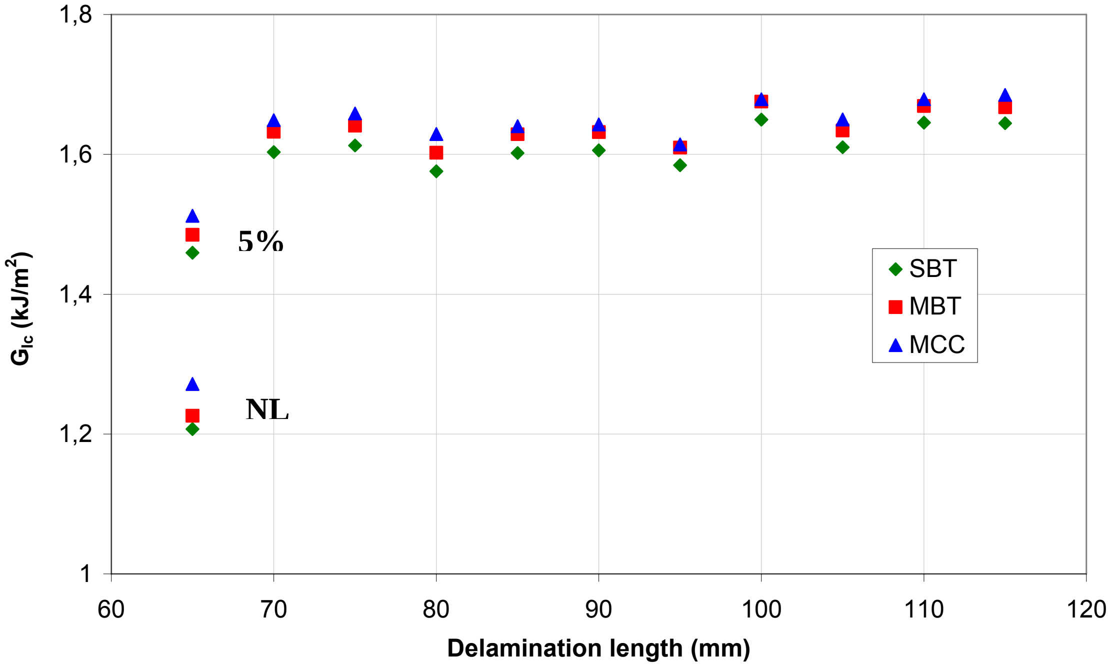

Figure 7: Delamination resistance curve (R curve) of a 6 bars consolidated specimen

4. EXPERIMENTAL RESULTS FOR S2-GLASS/PEEK PREPREG

4.1 Crack initiation

For each configuration (i.e. 6, 10, 20 and 30 bars), the NL and 5%/MAX initiation values of G_{Ic} are obtained by taking the average of the three specimens. For clarity reasons only the results obtained using the MCC method are presented here (figure 8).

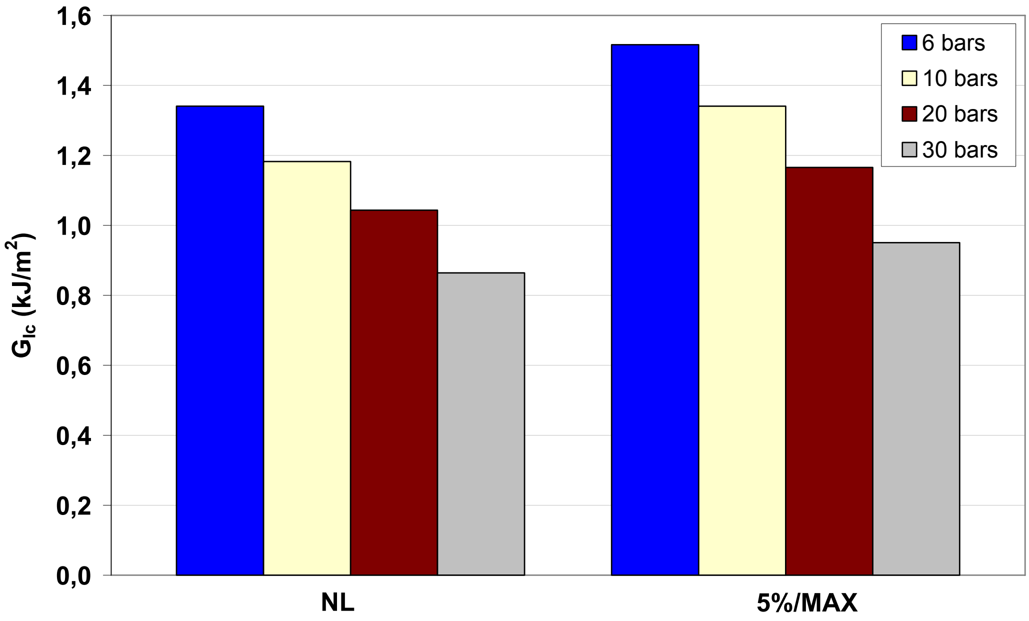

Figure 8: NL and 5%/MAX initiation values of G_{Ic} measured from the pre-crack

The obtained initiation values, NL and 5%/MAX alike, show very clearly that the higher the consolidating pressure is, the lower G_{Ic} is. Taking into consideration the ductility of PEEK, it is quite logical to observe that a crack will initiate less easily in a resin-rich zone than in a zone with low resin content. It can be noticed too that the 5%/MAX method systematically gives higher values than the NL method.

4.2 Crack propagation

For the specimens consolidated with the lowest pressures (6 -10 bars), the observations of the crack growth have revealed a very stable delamination process, resulting in smooth load-opening displacement curves (figure 6) and a very clean delamination surface (figure 9a). For one “20 bars” specimen and two “30 bars” specimens, a branching phenomenon has been observed: the decohesion of one of the two interface layers makes the delamination plan move from this interface to another one (figure 10). It results in uneven load-displacement curves showing an increased resistance to delamination as the crack grows (figure 11). The initial delamination surface shows a lighter zone corresponding to the secondary delamination plan (figure 9b).

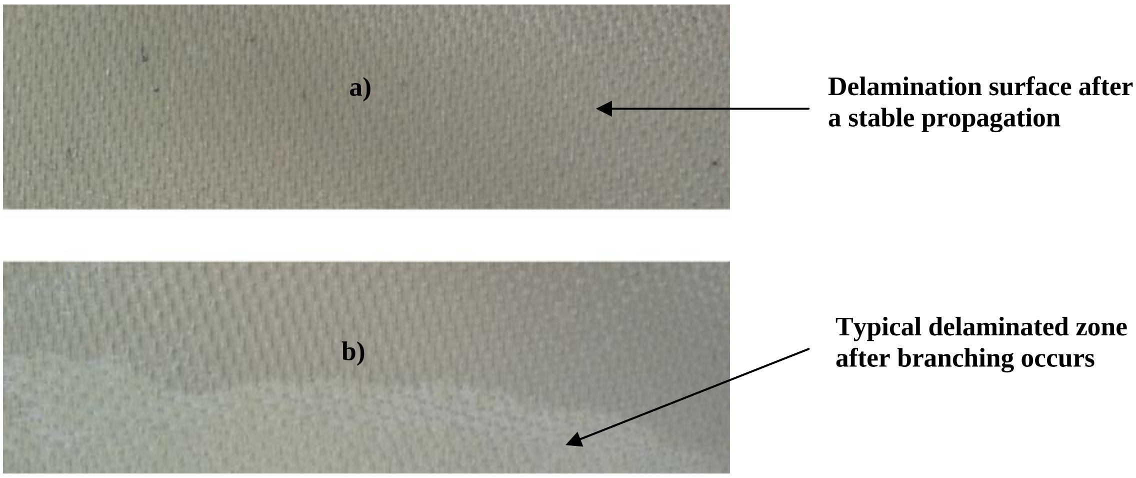

Figure 9: Delamination surfaces obtained for “6bars” (a) and “30 bars” (b) specimens

As the branching phenomenon isn't considered as representative of the specimen delamination resistance, the G_{Ic} propagation data of the concerned specimens weren't included in the calculation of the mean value. Even if the results obtained from the specimens whose delamination was stable were very homogeneous for each configuration (cf. figure 13), this point must be taken into consideration in the interpretation of these mean values.

Figure 10: Branching phenomenon observed for "20 bars" and "30 bars" specimens

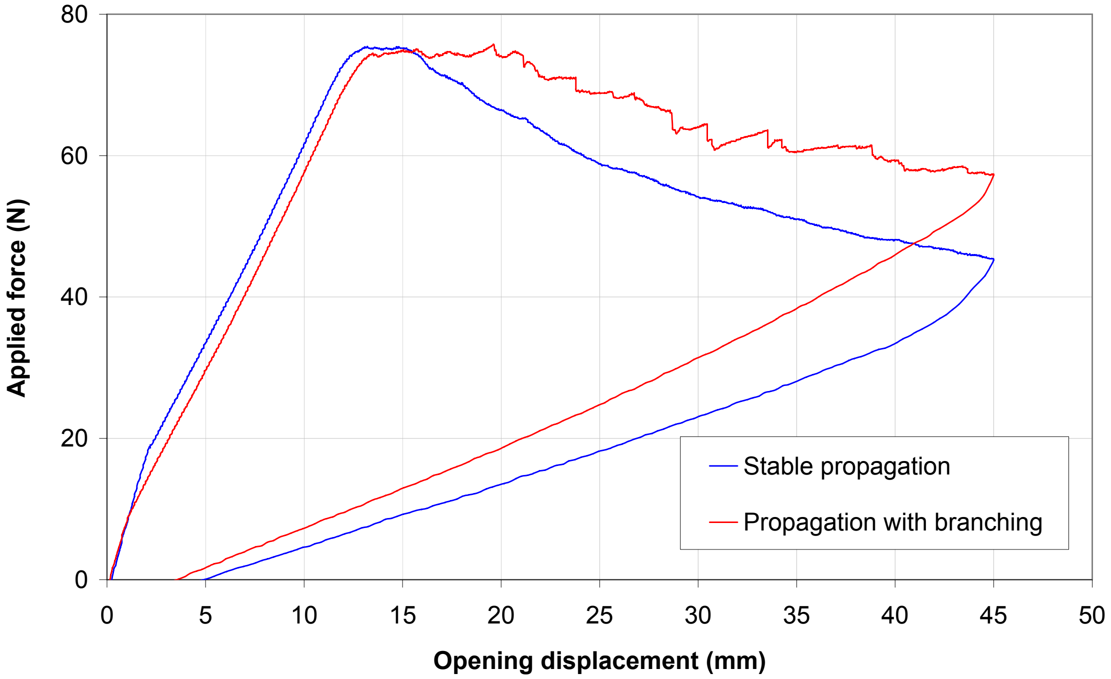

Figure 11: Comparison of load-displacement curves of "20 bars" specimens in case of a stable crack growth and in case branching occurs

The propagation value of G_{Ic} for each configuration and each data reduction method is presented below (figure 12). The general trend revealed by the initiation values is confirmed. The specimens consolidated with a pressure of 6 bars show the highest mode I interlaminar fracture toughness (>1.6kJ/m^2). The G_{Ic} values of the specimens consolidated with pressures of 10 and 20 bars are close to each other and much inferior to the "6 bars" specimens toughness. The G_{Ic} value decreases dramatically for the specimens consolidated with the maximal pressure of 30 bars. Among the three analysis schemes that were used, the MCC method gives the least conservative propagation value. The most conservative values are given by the SBT method.

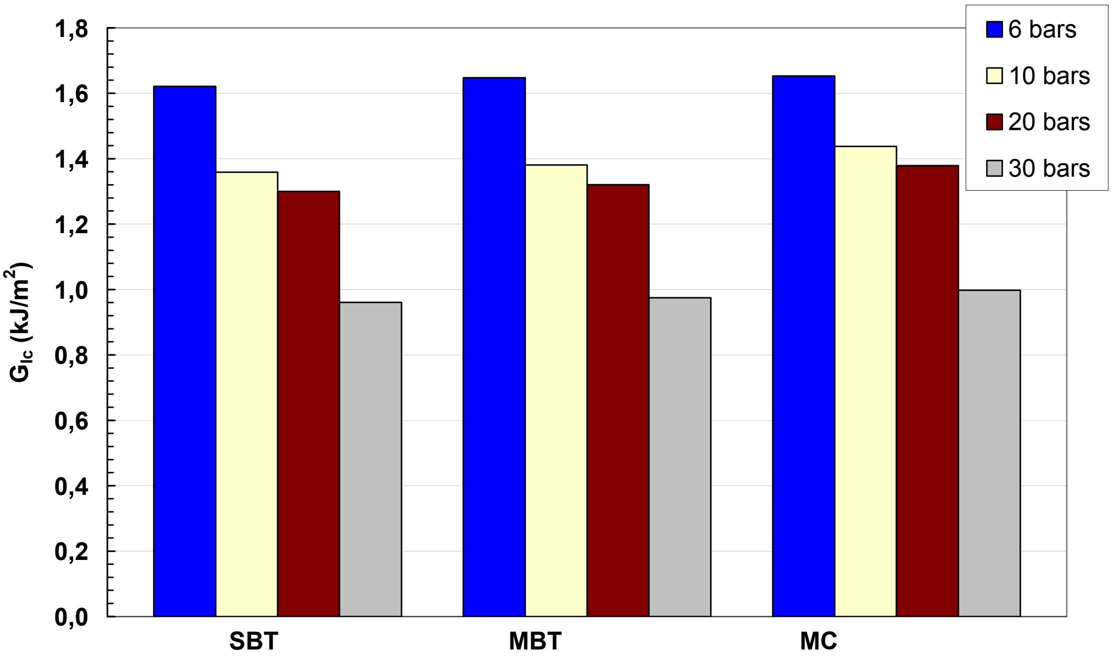

Figure 12: G_{Ic} propagation values calculated using the SBT, MBT and MCC methods

5. COMPARISON WITH E-GLASS/PEEK PREPREG

In order to investigate the influence of the reinforcement type, three DCB specimens were realised from the E-Glass/PEEK prepreg whose characteristics are presented in table 1. The manufacturing and testing processes were strictly identical to the previous ones. A consolidating pressure of 10 bars was used.

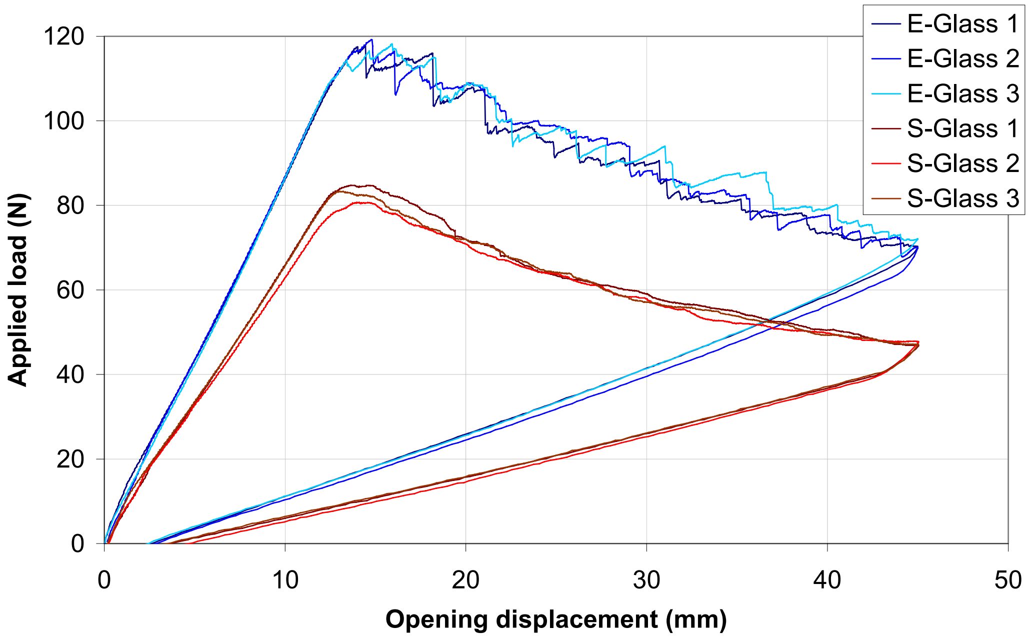

Figure 13: Load-displacement curves of the “10 bars” E and S2-Glass/PEEK specimens

The comparison of the load-displacement curves (figure 13) of both materials gives a lot of information. First, a much higher load is necessary to initiate and propagate the crack of the E-Glass reinforced specimens, what means they show a higher resistance to delamination. Further calculations based on the MCC method have lead to a G_{Ic} propagation mean value of 2.365 kJ/m^2, while the value obtained for the “10 bars” S2-

Glass reinforced specimens was 1.437 kJ/m^2. Secondly, the curves of the E-Glass reinforced specimens are much less smooth, what is indicative of less stable crack propagations. The observations made during the tests confirm that: the crack was jumping ahead abruptly as a visibly strong adherence between the two adjacent layers prevented from a stable separation. Bridging phenomena weren't observed but the examination of the specimens after the tests showed much rougher surfaces than in the case of the S2-glass reinforced specimens (figure 14).

Figure 14: Rough delamination surface of a "10 bars" E-Glass reinforced specimen

6. CONCLUSIONS AND PERSPECTIVES

This study has clearly shown that a higher resin content in the laminate leads to a better mode I interlaminar fracture toughness. Thus, the increased consolidating pressure wouldn't directly explain the improved resistance of the self-manufactured PEGASUS supporting structure. To confirm these results or question the relevance of G_{Ic} in the prediction of the delamination resistance of structures subjected to hyperacceleration, a E-Glass/PEEK based laminate consolidated with a low pressure will be built and accelerated with PEGASUS. The experimental data will be used to complete a Cohesive Zone (CZM) based model (not presented here for concision reasons) implemented in a finite element software for the simulation of the composite structure acceleration. In parallel, the influence of the cooling rate will be investigated by comparing the degree of crystallinity of both industrial and self-manufactured laminates.

REFERENCES

1- Reck B., Lehmann P., Wenning W., Vo M.D., "Study of new materials for the electromagnetic railgun PEGASUS payload supporting structure". ISL Report RV232, 2006.
2- Gao S.L., Kim J.K., "Cooling rate influences in carbon fibre/PEEK composites. Part 1. Crystallinity and interface adhesion", C*omposites: Part A*, 2000; 517-530.
3- Gao S.L., Kim J.K., "Cooling rate influences in carbon fibre/PEEK composites. Part 2. Interlaminar fracture toughness", C*omposites: Part A*, 2000; 763-774.
4- Lustiger A., Uralil F.S., Newaz G.M., "Processing and structural optimization of PEEK composites", *Polymer Composites*, 1990; 11.
5- AFNOR, "Détermination de la ténacité à la rupture interlaminaire en mode I de matériaux composites à matrice polymère renforcés de fibres unidirectionnelles", *NF ISO 15024*, 2002.
6- ASTM, "Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites", *D 5528-01*, 2001.