# INFLUENCE OF VOID CONTENT ON THE MECHANICAL PROPERTIES OF CARBON/PPS LAMINATES

J. Patou<sup>1</sup>, E. De Luycker<sup>2</sup> and G. Bernhart<sup>1</sup>

<sup>1</sup>Université de Toulouse, CNRS, Mines Albi, INSA, UPS, ISAE-SUPAERO, ICA (Institut Clément Ader), Campus Jarlard, F-81013 Albi, France julien.patou@mines-albi.fr, gerard.bernhart@mines-albi.fr

<sup>2</sup> Université de Toulouse, CNRS, Mines Albi, INSA, UPS, ISAE-SUPAERO, ICA (Institut Clément Ader), IUT Tarbes, 65016 Tarbes, France Emmanuel.De-Luycker@iut-tarbes.fr

**Keywords:** Thermoplastic, Void content, Powder impregnation, Prepreg, PPS, Consolidation, Autoclave

#### Abstract

Void content of several carbon fiber/PPS laminates was investigated to assess the impact of porosity level on mechanical properties of thermoplastic materials. Specimens with a  $[0^{\circ}/90^{\circ}]_{6}$  stacking sequence were produced by various autoclave and oven consolidation cycles in order to obtain a void content in the range of 2.0% to 6.0%. Mechanical properties (tensile strength and modulus; flexural strength and modulus, interlaminar shear strength) were measured and results were related to void content. Results show that contrary to tensile strength, flexural and interlaminar shear strength are particularly sensitive to void content. C/PPS tensile and flexural moduli are also weekly affected by void content.

## 1. Introduction

Use of thermoplastic resin systems for composite material processing offers significant advantages compared to thermoset ones as not shelf life and no volatile emissions. However, manufacturing thermoplastic materials undergoes limitations related to the high processing temperatures and the geometric complexity of the attempted parts.

Autoclave consolidation, widely used for thermoset composite manufacturing, is adapted to thermoplastic polymer composites if process conditions are well selected, i.e. consolidation pressure and temperature, vacuum level and consolidation time. Process parameters and consolidation cycles have an effect on mechanical properties and laminate material quality [1-3], but most of the results available in literature are related to thermoset based composites [4-8].

In order to investigate the impact of porosity level on mechanical properties for thermoplastic laminates, cycles were defined to obtain C/PPS sheets with a void content in the range of 2.0% to 6.0%. This paper addressed mainly the tensile, flexural and interlaminar shear properties in relation with consolidation conditions and porosity level.

## 2. Materials and experimental procedure

All laminates were produced using powdered 5H satin carbon fabric with PPS (Pipreg® from Porcher Industries). Semi-preg properties and morphology are given in Table 1 and Figure 1. Six layers of fabrics where stacked  $[0^{\circ}/90^{\circ}]_{6}$  and inserted between two Kovar plates in order to obtain laminates with flat surfaces.

Style	3106-1250P2353		
Weave	5 H Satin		
Warp / Weft	3K HS		
Dry fabric areal weight (g/m <sup>2</sup> )	285		
Polymer areal weight (g/m <sup>2</sup> )	216		

**Table 1.** Fabric properties



Figure 1.Carbon/fiber powdered fabric

Two processing equipments where used for the 400x300 mm<sup>2</sup> laminates manufacturing: an autoclave (SCHOLTZ for laminate N°1 and N°2) and an **oven** (Nabertherm for laminate N°3 and N°4). Processing conditions are detailed in Table 2, which reports the consolidation temperatures and times, as well as the sheets external pressure environment and internal vacuum condition. All sheets have a thickness of (2.0+/-0,1 mm).

Table 2. Laminate processing cycles

Laminate number	Consolidation temperature (°C)	Consolidation pressure (bar)	Vacuum level (mbar)	Consolidation time (min)	Pressure application condition
1	310	5	/	10	The whole process
2	310	5	150	10	Only during the consolidation phase
3	320	/	400	10	N/A
4	320	/	380	20	N/A

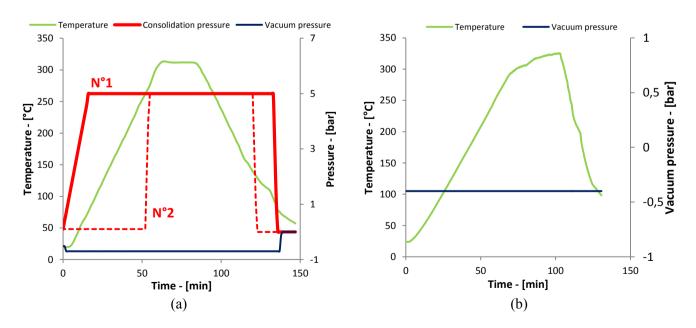


Figure 2. Processing cycles : (a) autoclave consolidation – (b) Oven consolidation for laminate N°4

Figure 2 shows two typical thermal cycles regitered with a thermocouple in the composite sheets. Autoclave temperature profile is close to the expected cycle as the temperature regulation thermocouple is on the mold. For oven consolidation, vacuum level (around -400 mbar) was maintained all along the cycle whereas it varied for the autoclave cycles (no vacuum or -150 mbar). External pressure application condition was also changed for the autoclave cycles as it is illustrated on (Figure 2(a)). A pressure of 5 bars was applied all along the cycle for laminate N°1 and only during consolidation phase for laminate N°2 when PPS reached its minimum viscosity.

### 2.2 Experimental procedures

The samples used for laminates characterization were cut by water jet cutting. In each composite part, five  $100x15x2 \text{ mm}^3$  flexural specimens, five of  $110x10x2 \text{ mm}^3$  compressive samples, eight  $250x25x2 \text{ mm}^3$  tensile samples and  $15 \ 20x10x2 \text{ mm}^3$  samples for interlaminar shear testing and porosity measurement were machined.

The void content level was obtained by matrix digestion in a concentrated sulfuric acid solution according to NF EN 2564. Three samples of each laminate were analyzed to get an acceptable average of the void content.

An optical microscope equipped with a camera was used to perform porosity characterization in terms of location and shape. Each sample was coated by EPOLAN 5015 resin, polished and observed at different magnifications: x25, x100 and x200. For each laminate, a minimum of two samples were cut to perform this analysis.

Mechanical tests were performed in accordance with the international standards: ISO 14130 for interlaminar shear, ISO 14125 for three point bending and ISO 527 for tensile tests. For each condition, five samples were tested except for the tensile 90° (only 3 samples).

#### 3. Results and discussion

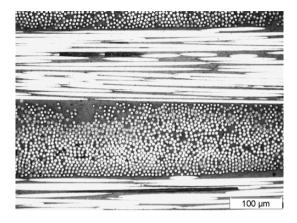
## 3.1 Influence of consolidation cycle on the porosity.

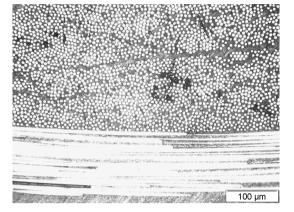
As reported in Table 3 all laminates have the same fiber volume fraction close to 50%. On the contrary void content varies from 2.4% to 5.8%. As expected maximal void content is measured on the sheet consolidated in an oven for 10 min. An increase of consolidation time decreases void content (5.8% down to 4.6% when moving from 10 to 20 min). Application of an autoclave pressure of 5 bars allows a significant decrease of void content and use of vacuum during autoclave forming increases laminate quality reaching aeronautical standards (<2.5%). Mean ply thickness is close to 0.323 mm, whatever the laminate (Table 3). Note that this thickness is slightly more important than the value given in Porcher data sheets (0.317 mm) obtained on sheets manufactured under mechanical presses.

Laminate number	Mean void content (%)	Fiber volume fraction (%)	Laminate thickness (mm)	Ply thickness (mm)
1	2.60	49.4	1.97	0.327
2	2.40	49.9	1.90	0.317
3	5.80	50.6	1.97	0.328
4	4.60	50.6	1.94	0.323

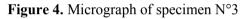
Table 3. Mean void content, fiber volume fraction, laminate and ply the	thickness
---	-----------

Each processing cycles described in Table 2 correspond to a specific void content. Processing parameters choice as applied pressure, consolidation temperature, vacuum level and consolidation time take a part on this phenomenon. Figure 2 and Figure 3 show two consolidation situation.





**Figure 3.** Micrograph of specimen N°2



Optical micrographs show no interply porosities; all are intra-ply voids and mainly located inside the fiber bundles as it can be seen in Figures 3& 4. Moreover is can be seen that consolidation pressure application limits the void formation inside the bundle as reported previously in literature [1]. In laminate N°2 with a void content of 2.4% there are small voids inside the bundle whereas for laminate N°3 with 5.80% the void are larger.

## 3.2 Mechanical properties of the C/PPS system in relation with void content .

The void content has a significant impact on the mechanical properties of a C/PPS laminate as it can be seen in Table 4.

Laminate number	Three point bending		ILS	Tensile – Warp (0°)		Tensile – Weft (90°)	
	Stength (MPa)	Modulus (MPa)	Shear stress (MPa)	Strength (MPa)	Modulus (MPa)	Strength (MPa)	Modulus (MPa)
1	673.8	49996.0	42.4	786.0	54322.7	730.2	54286.9
2	758	50843.0	45.4	731.4	57698.0	780.6	58724.0
3	449.47	47825.8	34.07	661.9	52739.6	683.8	52779.0
4	515.86	48032.5	38.25	720.6	55217.8	753.3	53597.4

**Table 4.** Mechanical properties of the laminates

As it can be seen in Figure 5, all strength properties are decreasing with the void content. However, the drop depends on the type of mechanical test. Flexural and interlaminar shear strengths vary linearly with void content with, respectively, a maximum value of 760 MPa and 46 MPa for 2.4% porosity. On the contrary tensile strengths are more or less constant up to a porosity level of 5%. Note that strengths values are higher for 90° (weft) direction than for 0° (warp) direction for all the laminates. Difference can reach 100 MPa. Standart deviation is also higer for tensile values.

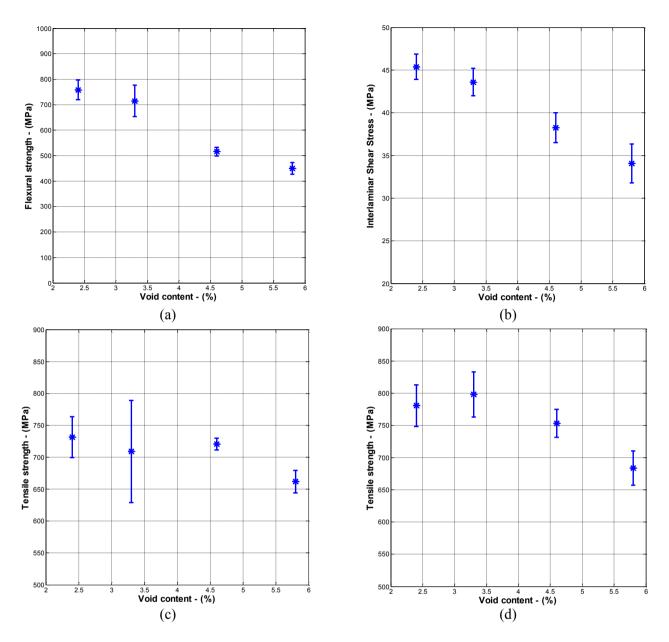
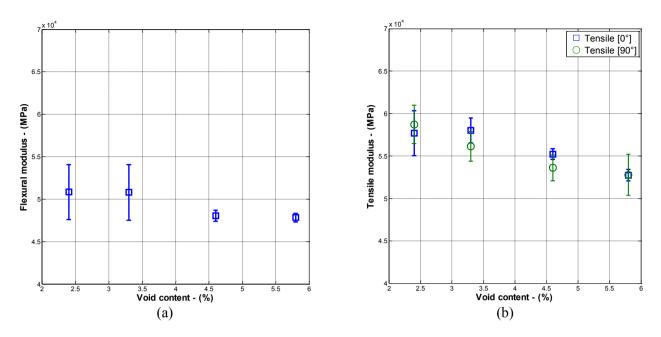


Figure 5. Effect of the void content on mechanical properties: a) Flexural strength b) ILSS c) tensile  $0^{\circ}$  (warp) d) tensile  $90^{\circ}$  (weft)

Flexural and tensile elastic moduli are plotted in Figure 6. Even if the high standard deviation for the lower void contents does not permit to establish a clear statement for flexural modulus, it seems that he is slightly decreasing when void content increases. Trend is more clear for tensile moduli in both testing direction (0° and 90°): elastic modulus decreases from 57700 MPa for 2.4% to 52700 MPa for 5.8%.



**Figure 6.** Elastic moduli: (a) Flexure – (b) Tensile

Relative variation of the strengths and the elastic moduli are presented in Figure 7. It can be seen that the interlaminar shear and flexural strength are the most sensitive to void content. The flexural strength decrease is close to 40% when the void content varies from 2.6% to 5.8%. The interlaminar shear strength falls by approximatively 20% for a void content of 5.8%. Flexural and ILSS test induce matrix shear stresses that are more sensitive to void content than tensile stresses (decrease limited to 10%) where mostly the fibers are loaded.

Elastic moduli decrease similarly in flexural and tensile configurations, limited to 10% as it can be seen in Figure 7(b). This can be explain by the high rigidity of carbon fiber which support the whole tensile stress even in a flexural configuration. Consequently the effect of porosity is lowered.

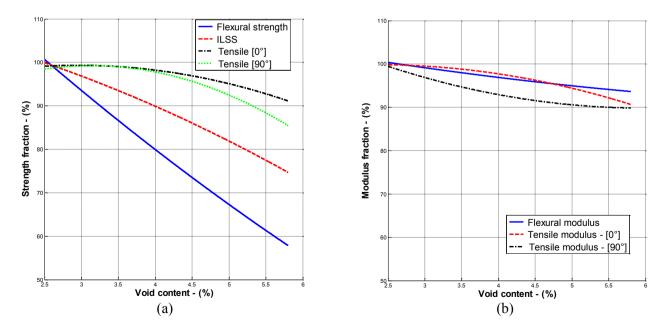


Figure 7. Relative mechanical properties variation : a) Strengths – b) Moduli

#### 4. Conclusions and perspectives.

Several C/PPS composite laminates were consolidated with different processing cycles to obtain a void content in the range of 2.0% to 6.0%. The experimental results show that the strength and modulus decrease with the increasing of void content whatever the nature of the mechanical stress. A higher sensitivity level has been noticed for interlaminar shear (25%) and flexural strength (40%) compared to tensile strength in warp and weft orientation (10%). Porosity has also a little impact on flexural and tensile moduli with a decrease limited to 10%.

Futher work will focus on compressive properties and a more detailed assessment of void distribution and sizes using tomography.

#### Acknowledgments

Authors acknowledge the financial support of BPI France and Region Midi-Pyrénées in the frame of ACAPULCO project.

#### References

- [1] A. Lystrup and T. L. Andersen, "Autoclave consolidation of fibre composites with a high temperature thermoplastic matrix," J. Mater. Process. Technol., vol. 77, pp. 80–85, 1998.
- [2] C. Santulli, R. Brooks, C. D. Rudd, and a C. Long, "Influence of microstructural voids on the mechanical and impact properties in commingled E-glass/polypropylene thermoplastic composites," *Proc. I MECH E Part L J. Mater. Appl.*, vol. 216, no. 2, pp. 85–100, 2002.
- [3] L. Ye, V. Klinkmuller, and K. Friedrich, "Impregnation and Consolidation in Composites Made of GF/PP Powder Impregnated Bundles," *J. Thermoplast. Compos. Mater.*, vol. 5, no. January, pp. 32–48, 1992.
- [4] H. ZHU, D. LI, D. ZHANG, B. WU, and Y. CHEN, "Influence of voids on interlaminar shear strength of carbon/epoxy fabric laminates," Trans. Nonferrous Met. Soc. China, vol. 19, pp. 470– 475, Sep. 2009.
- [5] L. Liu, B.-M. Zhang, D.-F. Wang, and Z.-J. Wu, "Effects of cure cycles on void content and mechanical properties of composite laminates," Compos. Struct., vol. 73, no. 3, pp. 303–309, Jun. 2006.
- [6] H. Koerber, J. Xavier, P.P. Camanho, Y.E. Essa, and F. Martín de la Escalera. High strain rate behaviour of 5-harness-satin weave fabric carbon-epoxy composite under compression and combined compression-shear loading. *International Journal of Solids and Structures*, 54:172– 182, 2015.
- [7] F.Y.C Boey, S.W. Lye,"Void reduction in autoclave processing of thermoset composites; Part1: High pressure effects on void reduction", Composite, vol. 23, no.4, July 1992
- [8] S. De Almeida, Z. Neto, « Effect of void content on the strength of composite laminates », Compos. Struct., vol. 28, pp. 139-148, 1994