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Compaction Behavior Of Out-of-Autoclave Prepreg Materials

Léonard Serrano[1, a)], Philippe Olivier[2, b)], Jacques Cinquin[3,c)]

[1]*Institut de Recherche Technologique Antoine de Saint Exupéry, 118 Route de Narbonne 31432 Toulouse*
Institut Clément Ader, 3 Rue Caroline Aigle, 31400 Toulouse
[2]*Institut Clément Ader, 3 Rue Caroline Aigle, 31400 Toulouse*
[3]*Airbus Group Innovations, 12 Rue Pasteur, 92150 Suresnes*

[a)] Corresponding author: leonard.serrano@irt-saintexupery.com
[b)] philippe.olivier@iut-tlse3.fr
[c)] jacques.cinquin@airbus.com

Abstract. The main challenges with composite parts manufacturing are related to the curing means, mainly autoclaves, the length of their cycles and their operating costs. In order to decrease this dependency, out of autoclave materials have been considered as a solution for high production rate parts such as spars, flaps, etc… However, most out-of-autoclave process do not possess the same maturity as their counterpart, especially concerning part quality[1]. Some pre-cure processes such as compaction and ply lay-up are usually less of a concern for autoclave manufacturing: the pressure applied during the cycle participates to reduce the potential defects (porosity caused by a poor quality lay-up, bad compaction, entrapped air or humidity…). For out-of-autoclave parts, those are crucial steps which may have many consequences on the final quality of the laminate[2]. In order to avoid this quality loss, those steps must be well understood.

INTRODUCTION

Composite parts for aerospace applications are mainly produced by lay-up using automated methods (ATL, AFP, FLU...), followed by compaction and cure in a pressure vessel called an autoclave. Autoclave manufacturing, however, is not completely defect-free and expensive. Drawbacks to autoclaves include the high capital investment required to obtain the equipment, the high cost of the nitrogen gas used to pressurize the vessel, poor energy efficiency, and long cycle times. Autoclaves are also size-limiting, restricting the dimensions of the part that can be produced to the inner diameter of the vessel. As it will be impossible to meet the growing demand for composite aircraft structures with autoclave cure methods, the industry is looking to out-of-autoclave (OOA) processing. However, while traditional autoclave processing involves high pressures that suppress formation of porosity, OOA methods supply a maximum pressure differential of 1 atm. Thus, implementation of low pressure processing methods requires a clear scientific understanding of the effects of process parameters on porosity and defects.

One of the most critical factors restricting composite part acceptance for aerospace applications is void content. Voids, or porosity, are open spaces within a composite part, which act as strength-limiting defects. A level of porosity below 2% is required for aerospace structures, rejecting parts with void content exceeding this value.

There are many studies examining the influence of porosity on part quality for autoclave processed composite parts[3,4]. Mechanical properties such as the interlaminar shear strength, material toughness and moisture uptake are greatly affected by void content. Olivier et al. investigated the influence of autoclave cure pressure on composite void content[5] (Figure 1). These results indicate that autoclave cure pressures exceeding 0.8 MPa will result in void free

parts, while lower cure pressures lead to the occurrence of ellipsoidal voids. The influence of those void contents on ILSS value was also studied by Olivier et al.[5] (Figure 2).

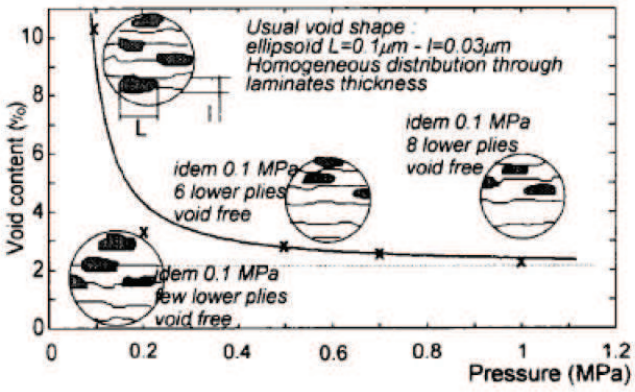

FIGURE 1. Void content as a function of pressure applied during process

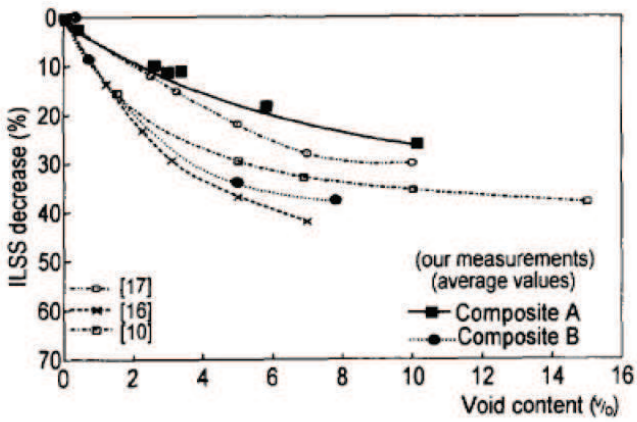

FIGURE 2. Interlaminar Shear Strength (ILSS) as a function of void content

In order to suppress voids and facilitate compaction, composite parts manufacturers have relied upon high autoclave pressures (up to 7 Bars or 0.7 MPa) applied during the cure cycles. With Out-of-Autoclave processing, however, the safeguards to void formation supplied by high cure pressures are removed. Thus, an understanding of the mechanisms of void formation is critical to successful defect reduction in low-pressure processed parts. The importance of pre-cure compaction process will also be studied in this document.

MATERIALS AND METHODS

The influence of pre-cure Compaction

Vacuum Bag Only process (or VBO) was selected as our Out-of-Autoclave processing way. The material used for this study was a UD prepreg made of a thermosetting matrix reinforced by IM7-12K carbon fibers. The curing temperature recommended by the material manufacturer was of 180°C. 4 plies of the prepreg were laid at 0° on a mold following the scheme on Figure 3. In order to study the pre-cure compaction, 5 samples were prepared with different compaction time under a constant vacuum of -0.08 MPa.

Those uncured samples are analyzed by X-Ray Tomography, under a resolution of 7µm per voxel, for a total scanned volume of 10x20x0.2 mm per sample. The porosity was measured by threshold on grayscale images having 256 level of light (0 to 255), following the Y Slices.

This porosity level calculation was made in python, computing the convex hull of the set of pixels in order to separate the pixels of the background from the pixels of the sample, binarizing the images with the threshold and calculating the proportion of pixels corresponding to voids in the sample. Those operations were repeated in the whole length of the sample in order to obtain an average value of porosity level (see Table 1)

FIGURE 3. Laminating configuration and ancillary products used for manufacturing samples

TABLE 1. Sample manufacturing conditions and configuration

Sample Name	Pre-cure Compaction Time (min)	Porosity Level (%)
C01	0	37.45
C02	30	25.02
C03	60	21.46
C04	120	15.42
C05	1440 (24h)	6.38

Porosity level during the cure cycle

We choose to follow the cure changes through rigidity evolution during the cure cycle, with the use of the Vanhograph machine[6].

The specimen of 50 x 75 mm is made of 7 plies oriented in this way [0° 90° 0° 90° 0° 90° 0°], the 0° are in the direction of the jaws in order to resist the holding of the specimen between the jaws, and making the cross lay-up in order to resist the holding between the jaws and the shear stress produced by the motor.

The resulting curve with the applied cure cycle is shown in figure 5, which can be separated into 4 domains:

- Domain 1: Classical evolution of viscosity with temperature increase. Objective is to have a compromise between minimum viscosity and maximum duration at minimum viscosity
- Domain 2 : Objective is to reduce porosity of the prepreg as the resin is very fluid, the dry fibers get easily soaked by the resin
- Domain 3: The objective is to produce the gelation of the resin, the transition between the liquid and solid state of the resin, when cross-linking exceeds a critical value (which depends only on the compounds of the resin)
- Domain 4: The objective is to completely polymerize the resin, which is the longest part because after gelation, the polymerization is controlled by diffusion of the molecules.

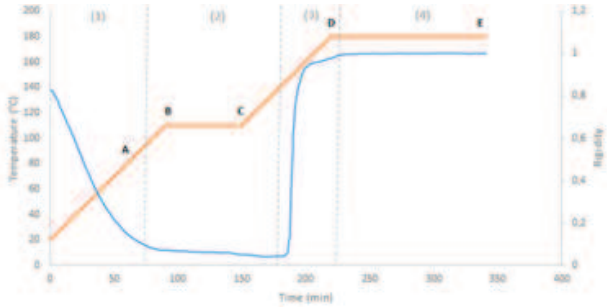

FIGURE 4. Vanhograph rigidity curve as a function of the cure cycle (Temperature, Time)

Five specimens were manufactured according to the configuration of Figure 3 with a lamination $[0°]_4$ each sample was compacted during 30 minutes before curing, and the cycle was stopped by thermal quenching, according to the points on Figure 4. The samples were analyzed by the same method (XR Tomography, 7 µm/Voxel, 10x20mm) as the previous test campaign. Porosity levels were obtained by following the same method as previously (see Table 2).

TABLE 2. Sample manufacturing conditions and porosity level

Sample Name	Sample width/length (mm)	Porosity Level (%)
C02	10/50	25.02
A	10/50	7.73
B	10/50	3.67
C	10/50	3.27
D	10/50	3.19
E	10/50	2.98

RESULTS AND DISCUSSIONS

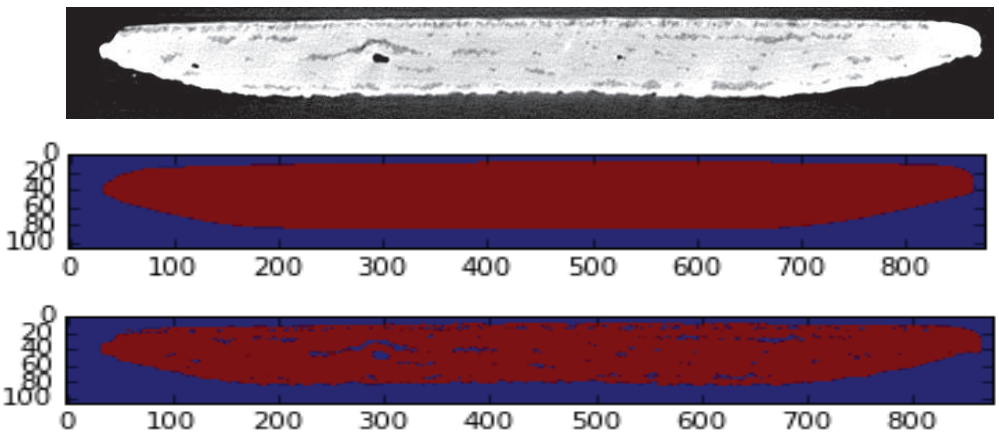

FIGURE 5. Tomography image of sample A (top); Convex Hull obtained with Python (middle) and binary image obtained by threshold (bottom).

As we can see in Figure 5, the porosities from entrapped air between layers (black), and porosities from dry fibers (darker grey) can be easily observed. The Convex Hull is accurate enough to allow us to make the calculation of the blue pixels in the bottom image (corresponding to the voids in the top image), without the pixels from the background. The grayscale contrast does not allow to separate the fiber and the matrix, a wider scale of gray is needed and longer acquisition times are required for this experiment.

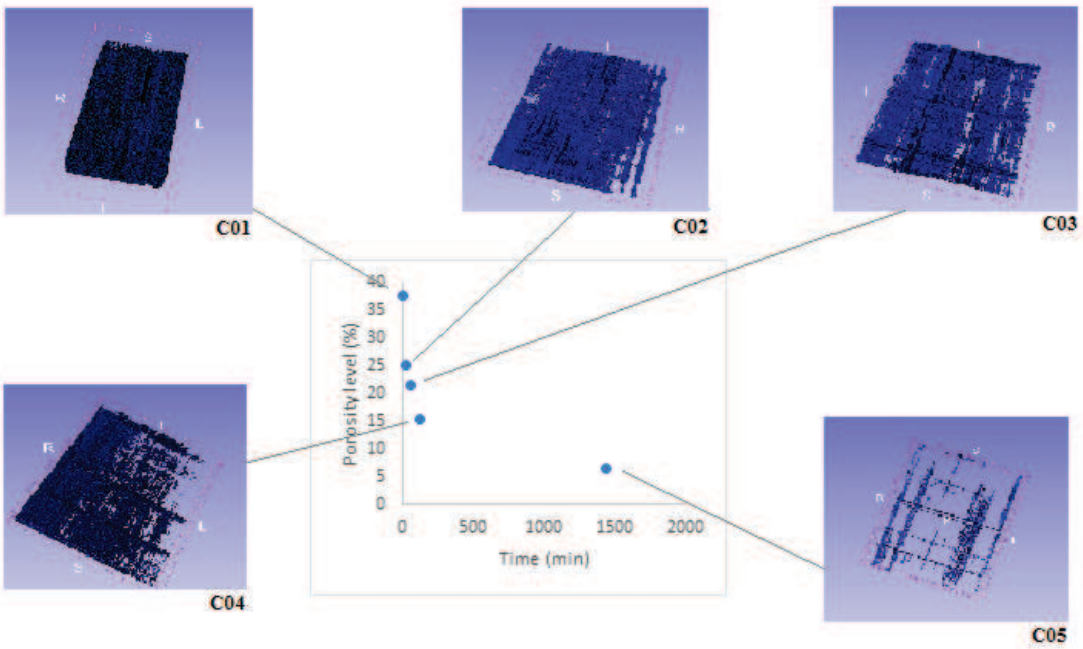

FIGURE 6. Porosity level as a function of the Compaction time and void representative pictures (Blue)

As expected, the porosity decreases with the compaction time (Figure 6), as air gets sucked away from the laminate, although, we can expect it to reach a minimum even after 24 hours, as the dry fibers will not get impregnated by the resin at room temperature, as a consequence, there will still be void inside the laminate before curing despite of the time spent for pre-cure compaction. In a further study, it would be interesting to observe the influence of the vacuum level and the thickness of the specimen on the porosity level, as in industrial production, composite parts are usually quite thick and the vacuum is distributed by inlets distant from each other.

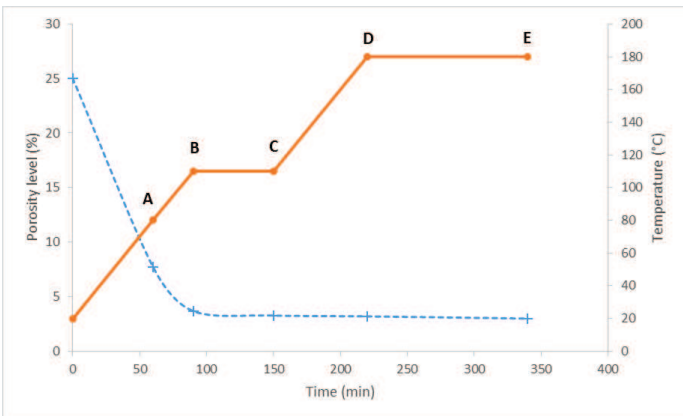

FIGURE 7. Porosity level as a function of the Cure cycle

As it was expected, the porosity decreases faster when applying heat to the laminate, as the viscosity decreases, the resin can move through the laminate, filling the voids and impregnating the dry fibers. The important decrease in void content occurs before the first dwelling step. It could be interesting to change the heat rate or the dwelling temperature and to observe its effect on the porosity level of laminates with the same configuration.

In spite of using the recommended cure cycle, we could only reach a porosity level of approximately 3%, which is constant after gelation as there is no movement of the matrix inside the laminate, when the degree of cure reaches a certain value, depending on the stoichiometry and functionalities of the reactants [7]. A complementary work will be based on the optimization of the cure cycle according to the evolution of rigidity and porosity levels that can be reached in order to comply with aeronautical exigencies.

SUMMARY

The influence of the pre-cure compaction time and cure cycle on porosity level were investigated. Void contributions were easily observed through the whole length of the specimen, although, because of the low difference in density between the fibers and the resin, we were not able to accurately separate one from another. We demonstrated that the pre-cure compaction plays an important part in the manufacturing of "void-free" laminates, which – we hypothesize – can even become crucial for thick laminates.

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