Revista de la Asociación Española de Materiales Compuestos
MATERIALES COMPUESTOS
http://revista.aemac.org

http://revista.aemac.org/
vol 2, nº 3, pág. 110-114

ISSN: 2531-0739

J. J. Torres[a], F. Sket[a], C. González[a,b], M. Simmons[c]

[a] Instituto IMDEA Materiales, C/Eric Kandel, 2, 28906 Getafe, Madrid, España
[b] Departamento de Ciencia de los Materiales, Universidad Politécnica de Madrid, C/ Prof. Aranguren, 3, 28040 Madrid, España
[c] Hexcel Composites, Duxford, Cambridge CB22 4QD, Reino Unido

Mecanismos de evacuación y evolución de poros en laminados procesados fuera de autoclave

RESUMEN

Historia del artículo:
Recibido 5 de Mayo 2017
En la versión revisada 5 de Mayo 2017
Aceptado 31 de Mayo 2017
Accesible online 21 de Junio 2017

Palabras clave:
Fuera de autoclave
Preimpregnado
Bolsa de vacío
Porosidad
Tomografía de rayos X

El objetivo de este estudio es entender el mecanismo de formación y transporte de poros, así como la evolución de la porosidad durante el proceso de curado del laminado fuera de autoclave. Los laminados fueron conformados mediante el apilamiento y compactación de preimpregnados mediante dos tecnologías diferentes: manual (Hand lay-up, HLU) y automática (Automated fiber placement, AFP). Ulteriormente fueron curados mediante la técnica de bolsa de vacío en un horno industrial a presión atmosférica. La caracterización de la porosidad se realizó mediante tecnologías de ultrasonidos y tomografía de rayos X en diferentes estados del ciclo de curado. Las inspecciones mediante ultrasonidos demostraron que la porosidad se encuentra heterogéneamente distribuida, con mayor fracción volumétrica de poros en el centro del laminado. La tomografía de rayos X proporcionó información en tres dimensiones (3D) de muestras de dichos laminados y permitió realizar un estudio cuantitativo y cualitativo de los poros dentro de los mazos de fibras y los localizados entre capas de preimpregnados durante el proceso de curado.

Los preimpregnados diseñados para fuera de autoclave están parcialmente impregnados de tal manera que proveen canales de evacuación de aire en los mazos de fibras. La porosidad de dichos canales fue eficientemente extraída y permitió que una fracción volumétrica de poros atrapados entre capas fuesen evacuados durante el proceso de curado. Se comparó el comportamiento de los canales de evacuación y la evolución de la porosidad para las dos tecnologías de apilamiento. Además se realizó un modelo analítico de la evolución de un poro.

Mechanisms of air removal and void development in Out-of-Autoclave processing of laminates

ABSTRACT

Keywords:
Out of Autoclave
Prepregs
Vaccum bag only
Porosity
X-ray tomography

This study aim at understanding the void formation, transport mechanisms and porosity evolution during OoA consolidation process. The laminates were conformed by stacking and compacting of prepregs by two different technologies: manual (Hand lay-up, HLU) and automatic (Automated fiber placement, AFP). Subsequently the laminates were cured by the vacuum bag only (VBO) technique in an industrial oven at atmospheric pressure. The porosity characterization was performed using ultrasound and x-ray tomography technologies at different stages of the cure cycle. Ultrasonic inspections showed that the porosity is heterogeneously distributed, where the higher volumetric fraction of pores is located in the center of the laminate. X-ray tomography provided three-dimensional (3D) information of samples of the laminates and it allowed a quantitative and qualitative study of the pores within the tows of fibers and the porosity located between the layers of prepregs during the curing process.

The prepregs designed for out of autoclave are partially impregnated in such a way to provide air evacuation channels in the fiber tows. The porosity of these channels was efficiently extracted and a volumetric fraction of pores entrapped between layers were evacuated during the curing process. The behavior of the evacuation channels and the porosity evolution for both stacking technologies were compared. An analytical model of the evolution of a pore was also developed.

Autor de contacto: Juan Jose Torres
e-mail: juanjose.torres@imdea.org

Año 2018

1 Introduction

During the last decades, structural composite parts of aircrafts were manufactured by the autoclave consolidation of laminates formed by stacking pre-impregnated carbon sheets (prepregs). However, autoclave consolidation requires large capital investments. Therefore, significant efforts were invested in out-of-autoclave techniques (OoA) aiming at maintaining autoclave quality. To this end, OoA prepregs are specifically designed to meet the low porosity levels required in the final parts (<2%), when cured only under vacuum pressure and standard industrial ovens.

Several studies, e.g. [1, 2], have shown the negative effect of porosity on the mechanical properties (e.g. fracture toughness, compression strength, inter-laminar and in-plane shear strengths) of fiber reinforced polymers (FRP). Autoclave prepregs usually consist of unidirectional (UD) or woven fabric which are completely filled with resin. Thus, when applying the cure cycle under high hydrostatic pressures (in the order of 7bar), the dry areas are filled by resin and the entrapped air or volatiles are collapsed or removed, thus reducing the void content and providing free porosity panels. However, in VBO processing, the applied pressure during curing can reach a maximum value of 1 bar from the applied vacuum, making the resin flow and void evacuation more difficult. The resin pressure depends on the compaction pressure, which is not enough to collapse the voids in VBO process.

A proper design of prepregs requires taking into account the processes occurring during the whole manufacturing procedure, from lay up until the final curing. Therefore, OoA prepregs are designed with a different approach. OoA prepregs consist of partially infiltrated tows aiming at providing adequate evacuation channels for the voids. This architecture creates air channels intended for entrapped air to escape due to the application of vacuum and resin to flow into the channels, filling them and providing a high quality panel. Thorfinnson et al. [3] investigated processing parameters that affect voids content in unitape prepreg structures. They explained the dependency of gas permeability with resin impregnation degree and found out that partially impregnated prepregs provided voids free parts due to the evacuation paths in the prepregs. Therefore, the combination of the resin viscosity and kinetics with the void evacuation channels was proved to be an efficient technique to provide the almost free void parts, at least in relatively thin parts. Centea et al. [4] have carried out a study on sequencially cured laminates to study the evolution of porosity (evaluating the void percent of different types of voids), concluding that the tomography is a powerful technique to evaluate the microstructure of prepregs and they quantified the filling resin in the tows during the cure cycle and the entrapped air decreases until the tows are fully impregnated, then the air evacuation ceased. Also, Centea et al. [5] developed a model to simulate the transverse impregnation of dry fibre tows within of OoA prepreg for any cure cycle.

2 Methodology

2.1 Materials and laminate preparation and processing

The prepreg material used in this study is HexPly M56 prepregs (commercially available and supplied by Hexcel). HexPly M56 is a high performance epoxy matrix developed for out-of-autoclave curing for composite aircraft structures. In prepregs used in this work the matrix is reinforced with IM7. Flat panels of 400x400mm were produced by stacking 24 plies of unidirectional prepregs with a [+45/0/-45/90]3s stacking sequence. The stacking procedure is critical to minimize the amount of entrapped air. For the HLU procedure, the debulking was conducted for each four plies for 10 min. Once all plies are stacked, the edges were cut in order to re-open the evacuation channels. Regarding the AFP laminates, the stacking procedure was carried out by AFP machine and the values of the parameters used in the process are not known. All laminates were cured with the VBO, taking into account that in OoA prepregs, excessive debulking may be detrimental for void removal, since the in-plane gas permeability decreases with debulking time [6] and the debulking pressure might compress the tows and close the air channels for voids and volatiles evacuation [7, 8].

The microstructure of the laminates was studied at different cured stages. Figure 1 and table 1 show the applied cure cycle and the conditions where the cure cycle was stopped to produce partially cured panels. The processing windows, i.e. the stage during the cure cycle were the resin viscosity remains sufficiently low to allow void evacuation, is considered to start between time t0 and t1 and finish between time t2 and t3. The AFP laminates were also cured with VBO technique, however these were cured under modified cure cycle in order to avoid the delay between the oven and laminate temperature due to the thermal inertia. Therefore the time of the first dwell was modified to 90 min.

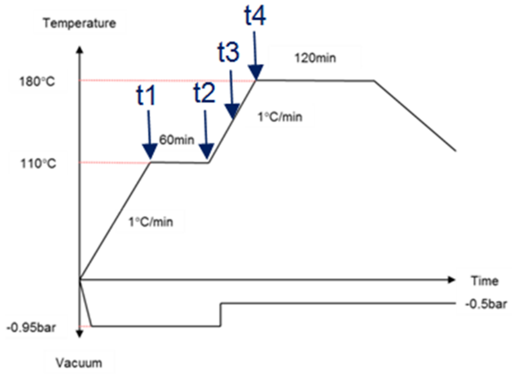

Figure 1. Applied cure cycle and stages for sequential evaluation of void evolution

Stage	Time (min)	Temp (ºC)
t0 (Initial)	0	20
t1	90	110
t2	150	110
t3	185	145
t4	220	180
t5 (Final)	400	20

Table 1. Time and temperature for each stage during the cure cycle

2.2 Tomography procedure

XCT (Nanotom 160NF Phoenix tomograph) was used to study the internal porosity of the laminates for each stage of curing (t0-t5). Sample coupons of 20x20mm were extracted from the center and near the panel edge. The coupons were measured at 100kV and 170µA with an exposure time of 0.75sec, and the obtained voxel size was 23µm. The reconstructed 3D volumes were segmented to separate different pores from the material.

3 Results and discussion

3.1 Inter-ply and intra-tow porosity at different stages of HLU laminates

The porosity within the tows was evaluated from the volumes and yielded a volume fraction of pores of 10 - 12% in the fresh panel (t0). The samples studied were extracted from the center of the panels, since the C-scan inspections showed that the porosity in the overall of the panels follows a radial distribution, where the highest values of the attenuation were concentrated in that region. Figure 2 shows the 3D porosity distribution of an uncured (t0) and the Figure 3 the cured coupon (t5). The uncured laminate contains flat-shaped voids corresponding to the inter-ply entrapped air as well as large amount of voids in dry fibers regions of the tows. In the fully cured panel, the void orientation follows the fiber direction. The majority of the remaining pores after cure are located at the ply interfaces. Most of the air within the tows was removed.

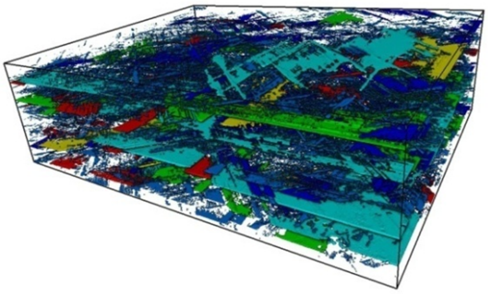

Figure 2. Voids distribution of uncured HLU laminate sample

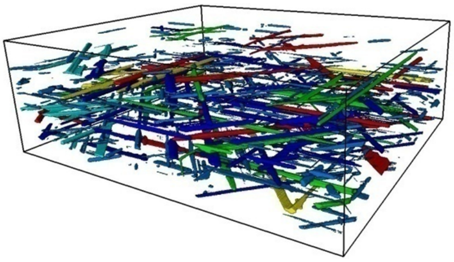

Figure 3. Voids distribution of cured HLU laminate sample

Tomographies of the samples at the four selected curing stages were segmented in order to obtain the inter-ply and intra-tow voids. The evaluated values of the void volume fraction of the inter-plies and intra-tows for each stage are shown in Table 2. The intra-tow porosity decreases to almost zero while the inter-ply porosity changes but the initial and final void content are similar. Also, the inter-ply voids increase after the first dwell probably due to pore expansion in the second temperature ramp.

Step	Time (min)	Temp (°C)	Inter-ply voids (%)	Intra-tow voids (%)	Degree of cure (%)
t0 (Initial)	0	20	1,9	4.2	26
t1	90	110	0,6	2.7	31
t2	150	110	0,4	0,5	36
t3	185	145	0,8	0	50
t4	220	180	2.1	0	93
t5 (Final)	400	20	1.6	0	100

Table 2. Voids percent in the interface between plies, within tows and the degree of cure for each curing stage

3.2 Inter-ply and intra-tow porosity at different stages of AFP laminates

Regarding the AFP laminates the pathways also work perfectly and the inter-ply porosity is less than in the HLU laminates, therefore this porosity is extracted by the evacuation channels easily. Regarding the initial conditions, the main differences between the HLU and AFP laminates is the initial material, therefore the variables to take into a count are the void volume fraction, shape and size of the voids and the dry region of the tows, which depends of the compaction pressure and temperature during the AFP process. The results were better than expected, since from the second up to the final stage the amount of pores were similar and negligible, for that reason in this work the results of the third and fourth stages are not shown.

A C-scan inspection of the semi cured laminates from the initial to the t2 stages (Figure 4) showed the same conclusion obtained by the tomographies. The ultrasound images showed that the initial and t1 stages present higher attenuation of the waves than the t2 stage. It is due to the dry region of the tows (pathways) which are not closed up to the second stages.

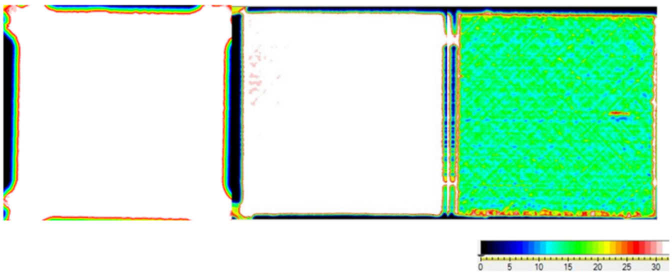

Figure 4. C-scan of AFP laminates, Initial, t1 and t2 stages

At the initial stage the void volume fraction without count the pathways is about 4%. Figure 5 shows a reconstructed volume of a sample of AFP laminate at the initial stage, where the pathways and voids are segmented. The amount of pores is reduce up to 0.01% from the initial to the first stage, since inter-ply and intra-ply voids have been evacuated, therefore the pathways from initial stage to t1 work perfectly and this interval is critical. The pathways remain open but the resin has filled a part of the tows. At the t2 stage the void volume fraction remain around 0.01%, the tows are completely filled and the sample does not show porosity, therefore the dwell time of 1h30' is enough, maybe this processing windows time can be reduce. At final stage (Figure 6) the laminate presents a high quality since the void volume fraction is about 0.14%.

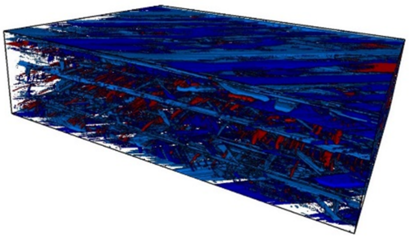

Figure 5. Voids distribution of uncured AFP laminate sample

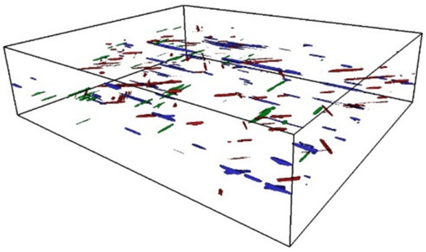

Figure 6. Voids distribution of cured AFP laminate sample

3.3 Evolution of intra-ply voids in OoAprepregs by analytic model

Porosity within the plies is essentially composed of intra-tow voids. Assuming that intra-tow voids are open cylinders following the fiber direction and connected to the vacuum ports, these voids should collapse primarily by radial resin flow. Figure 7 summarize the representative geometry of the model developed. The outer surface of the representative void element is subjected to the atmospheric pressure patm transmitted through the vacuum bag. The inner surface is connected to the vacuum pressure pvac and represents a typical intra-ply channel observed in this partially impregnated material.

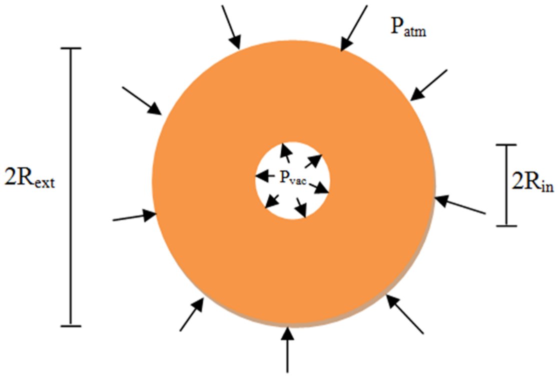

Figure 7. Representative volume containing a intraply void in OoA prepreg

By taking into consideration the initial volume fraction, the incompressibility of the fluid and the Darcy's law, it is possible to develop an expression that represents the time evolution of the internal radius of a void decreasing from R_{int} to the final collapse. The void radius evolution depends on the permeability factor K, the viscosity of the resin, and the pressure gradient p_{atm}-p_{vac}. The expression is given in Equation 1:

$$-\frac{K}{\mu}(p_{atm}-p_{vac})t = \frac{1}{4}{R_{int}}^2\left(1+2\ln\frac{R_{ext}}{R_{int}}\right)-\frac{1}{4}V_0{R_{ext}}^2(1+2\ln\frac{1}{V_0}) \qquad (1)$$

To solve this problem, we have estimated some values from the XCT experiments (R_{ext} = 300µm, V_0 = 5.4%, vaccum pressure = -0.94bar) and others from the literature (µ = 770Pas, K = 3.7E-15m^2), and the experimental and theoretical void evolution were very similar.

4 Conclusions

Evacuation of entrapped air or volatiles through the channels within the tows requires a long enough processing windows since the movement of voids is slow due to the low pressure gradient and compaction pressure. It should be noticed, thought, that these laminates are composed of 24 layers and they can represent a real parts of aircraft structures. The high panel thickness presents a relevant difficulty and further complicates the extraction of all entrapped air.

The final porosity in HLU panels is mainly inter-ply porosity and depends mainly on the debulking process and the operator experience (the entrapped air has its origin in the hand lay-up process). The inter-ply void volume fraction in HLU panels evolves during the cure cycle and their mobility under pressure gradient is lower than intra-tow voids. On the other hand, the intra-tow porosity decrease almost completely, thanks to the very efficient pathways of the OoA prepregs. The channels in the tows evacuate practically all the porosity up to the end of the first dwell (t2). There is no connection between tows channels and inter-ply voids. In this kind of laminates the critical interval is from t1 to t2.

Regarding the laminates fabricated by AFP process, at the initial stage the laminate has a moderate amount of voids which are evacuated without any problem by the pathways. The interval from Initial to t1 is critical, since the Inter-ply and intra-ply voids (not counting due to the tows) is evacuated from the initial to the t1 stage. Surely the cure cycle time can be reduce in order to optimize the process, since the influence of the processing window for the AFP laminates is lower than for the HLU laminates. After extracting almost completely the entrapped air, the evacuation channels are perfectly closed at the stage 2. Once the AFP laminate is cured it presents high quality.

The results of the analytical model and the experimental results about the volume fraction of intra-tow voids have a similar behavior. The experimental evolution of the intra-tow pores is consistent with a radial viscous flow of the resin to the evacuation channels. Therefore, this model can provide a first approach of the intra-void evolution for different initial parameters.

Acknowledgments

The author gratefully acknowledge the financial support of the Hexcel company within the "AROOA" project.

References

[1] Costa ML, Almeida SFM, Rezende MC. The Influence of Porosity on the Interlaminar Shear Strength of Carbon/Epoxy and Carbon/Bismaleimide Fabric Laminates. *Compos Sci Technol*, **61(14)**: p. 2101 - 2108. (2001).

[2] Liu L, Zhang BM, Wang DF, Wu ZJ. Effects of Cure Cycles on Void Content and Mechanical Properties of Composite Laminates. *COMPOS STRUCT*. **73(3)**: p. 303 - 30. (2006).

[3] Thorfinnson B, Biermann T. Degree of Impregnation of prepregs - effects on porosity. (1987).

[4] Centea T, Hubert P. Measuring the impregnation of an out-of-autoclave prepreg by micro-CT. *Compos Sci Technol* **71**: 593–599. (2011).

[5] Centea T, Hubert P. Experimental observation and analytical modelling of the resin flow inside an out-of-autoclave prepreg. *10th International Conference on Flow Processes in Composite Materials (FPCM10).* (2010).

[6] Louis BM. Gas transport in out-of-autoclave prepreg laminates. (2007).

[7] Danzi M. Out of autoclave processing; Advanced Composite processing.

[8] Thorfinnson B, Biermann T. Production of void free composite parts without debulking. *Proc 31st int SAMPE symp*. p. 10. (1986).