MECHANISMS OF AIR REMOVAL AND VOID DEVELOPMENT IN OUT-OF-AUTOCLAVE PROCESSING OF HAND LAY-UP LAMINATES

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Abstract

The aim of this study is to understand the void formation, transport mechanisms and porosity evolution during OoA consolidation process in laminates produced by hand lay-up (HLU) and cured in an industrial oven, using vacuum bag only (VBO) technique. To this end, ultrasound (C-scan) and X-ray computed tomography (XCT) inspections were used to characterize the final void content and the void evolution, respectively. C-scan inspections show that the voids were heterogeneously distributed within the panel, being the higher values located at the center of the panel. XCT experiments were carried out sequentially at different curing times. These inspections showed that initial porosity in OoA laminates consists of intra-tow voids and inter-layer voids, which evolve during the cure cycle. Improvement and optimization strategies should be developed to improve panel quality. Detailed information was extracted from the XCT tomograms revealing that intra-tow voids were effectively removed during the processing window of the cure cycle, while inter-layer voids were not completely removed. An analytical model of intra-ply void evolution was developed and agrees well with the experimental XCT results.

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

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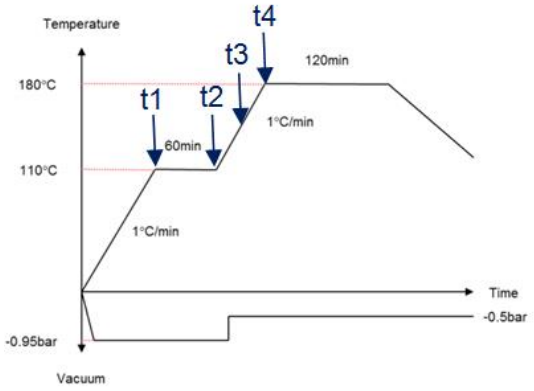
with resin impregnation degree and found out that partially impregnated prepregs provided voids free parts due to the evacuation paths in the prepregs. Therefore, the combination of the resin viscosity and kinetics with the void evacuation channels was proved to be an efficient technique to provide the 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 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 HLU laminates was studied at different cured stages. Figure 1 shows 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.



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

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

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 170uA with an exposure time of 0.75sec, and the obtained voxel size was 23um. 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

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). Figure 2 shows the 3D porosity distribution of an uncured (t0) and

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cured coupon (t5). The uncured laminate (Figure 2A) 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 (Figure 2B), 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.

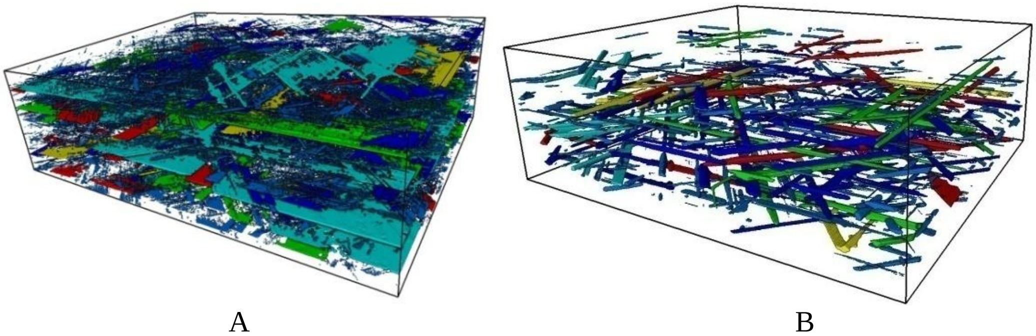

Figure 2. A) Voids distribution of uncured and B) cured HLU laminate sample

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 1. 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.

Table 1. Voids percent in the interface between plies and within tows for each curing state

Step	Time (min)	Temp (ºC)	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

3.2. Evolution of intra-ply voids in OoAprepregs by analytic model

Porosity within the plies is essentially composed of intra-tow voids. Assuming that intra-ply voids are open cylinders following the fiber direction and connected to the vacuum ports, these voids should collapse primarily by radial resin flow. Figure 3 summarize the representative geometry of the model developed. The outer surface of the representative void element is subjected to the atmospheric pressure p_{atm} transmitted through the vacuum bag. The inner surface is connected to the vacuum pressure p_{vac} and represents a typical intra-ply channel observed in this partially impregnated material.

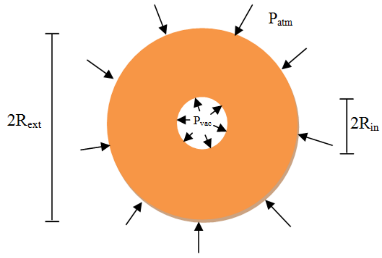

Figure 3. Representative volume containing a intraply void in OoA prepreg

By taking into consideration the initial volume fraction, the incompressibility of the fluid and the Darcyôs law, it is possible to develop an expression that represents the time evolution of the internal

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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 Eq. 1:

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

Eq. 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). With these the parameters the experimental and theoretical void evolution are 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. The volume fraction evolution of intra-tow voids obtained from the analytical model and the experimental results show similar behavior.

Acknowledgments

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