INFLUENCE OF PROCESS PARAMETERS ON THE QUALITY OF CARBON/PEKK LAMINATES MANUFACTURED BY OUT-OF-AUTOCLAVE CONSOLIDATION

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Abstract

Out-of-autoclave manufacturing of thermoplastic composites is one of the solutions to increase the production rate in the aeronautic industry. We present the identification of the process parameters to obtain a good consolidation quality of laminates. Different process conditions have been tested to produce unidirectional PEKK/cabon fibers laminates. The quality of the interlaminar consolidation of laminates has been evaluated by InterLaminar Shear Strength (ILSS) tests. Two principal process parameters which influence the interlaminar consolidation have been identified by the results of these tests: the compaction pressure and the maximum temperature reached during the thermal consolidation cycle. A monitoring technique of the laminates manufacturing has been developed to measure the temperature field through the thickness direction of laminate and the variation of the laminate thickness during the consolidation cycle. This monitoring system identifies two major consolidation phenomena: the one at the glass transition temperature (Tg) and the other at the melting point (Tm). These phenomena can be characterized by the difference of temperature peaks. At the Tg, the polymer molecule chains in amorphous configuration take part in the establishment of a contact between the adjacent layers.

1. Introduction

Thermoplastic matrix composites exhibit a lot of advantages over their thermoset counterparts such as high impact resistance and short process cycle time. Recently new high performance thermoplastic composites such as carbon/PEKK or carbon/PPS are attracting great attention from the aeronautic industry as cost effective alternatives to conventional carbon/PEEK composites. Another way to reduce manufacturing cost would be to develop out-of-autoclave processes where relatively low consolidation pressure is adopted. In this article we focus on low pressure consolidation of unidirectional carbon/PEKK laminates and in particular on the influence of process parameters on the quality of laminate consolidation. Indeed the different phenomena of laminate consolidation process (intimate contact [1–4], autohesion [5–8]) have been already investigated in the case of high pressure process whereas there are still some difficulties to explain the influence of a low pressure (or of an applied vacuum) on these phenomena [9, 10]. Firstly we investigate the influence of the process parameters as compaction pressure or cooling rate on the consolidation quality which has been evaluated including by mechanical tests (ILSS). Secondly we present an experimental system to monitor the evolution of laminate consolidation by measuring the temperature field and the laminate thickness.

2. Study of process parameters

2.1. Experimental method

We manufactured 16-UD-layers laminates of carbon fiber/PEKK prepregs in oven by applying compaction pressure. The process conditions are presented in Fig. 1. The compaction pressure was increased to a maximum value which was kept constant during the whole consolidation cycle. Whereas the same maximum temperature of 360°C was maintained for 15 minutes, different heating and cooling rates were tested. The process parameters which have been studied are the compaction pressure, the cooling rate, and the integral time/temperature during the overshoot at the thermal plateau.

The quality of interlaminar consolidation was evaluated by ILSS tests. The dimensions of specimens were 25×12×2.4 mm^3 where the greatest dimension corresponded to the fiber direction. The tests were performed on a Zwick/Roell Z010 with a load cell of 10kN.

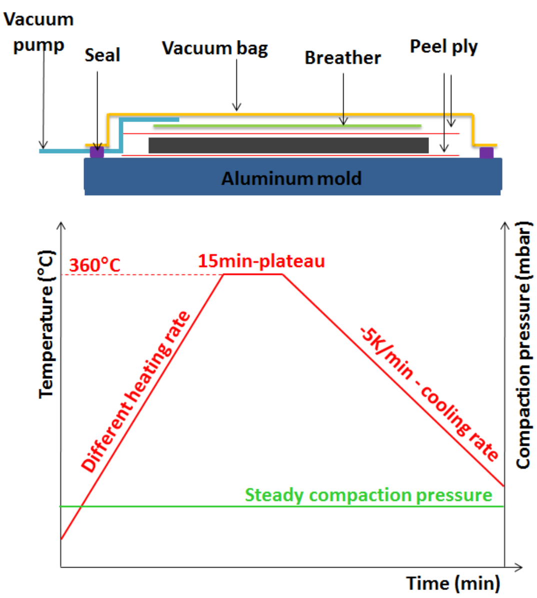

Figure 1. Manufacturing set-up (above) and process cycle (bottom)

2.2. Results and discussion

For the same thermal cycle different compaction pressures between 0mbar and 1000mbar were tested. A strong influence of the pressure on the consolidation quality was found (Fig. 2a). Above 700mbar a good consolidation was established between the adjacent plies of the laminate. Indeed the interlaminar shear strength reached almost 100MPa which was close to the value of a laminate consolidated under press (110 MPa). On the other hand, microscopic observations showed very few porosities inside the laminate and a good distribution of the fibers in the laminate. Thus, under certain conditions, laminates with high mechanical properties could be produced by out-of-autoclave process.

Under the high pressure (above 750 mbar), different cooling rates between -0.5K/min and -10K/min were tested to identify the influence of cooling speed on the interlaminar consolidation. Indeed, during the cooling phase, the crystallization of the PEKK which is a semi-crystalline matrix, takes place. The results of these tests showed that cooling rate in this range of rate had no influence on the quality of interlaminar consolidation (Fig. 2b). All the samples fabricated according to these conditions had a high interlaminar shear strength around 100MPa.

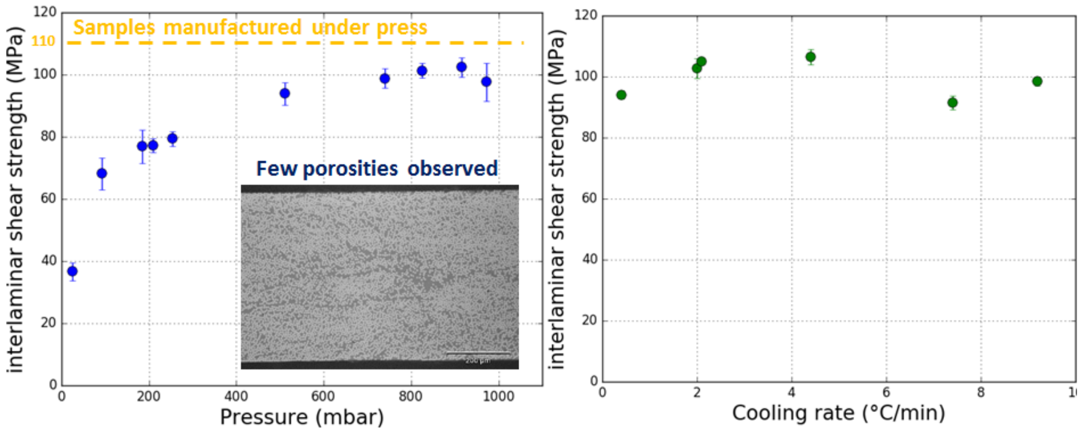

Figure 2. (a) Influence of the compaction pressure and (b) of cooling rate on the interlaminar consolidation

The influence of heating rate was also investigated. In particular, the change of heating rate modified the overshoot phenomenon (see Fig.3). An increase of the heating rate brought about a rise of the maximum temperature and a delay to reach this temperature. For the maximum heating rate used (20K/min) the maximum temperature value was around 7°C above the target temperature and remained above this temperature during about 4min. An evaluation of the interlaminar shear strength in terms of the integral time-temperature during this overshoot-phenomenon revealed that this temperature overshoot had a very high impact on the consolidation quality. For example, the interlaminar shear strength varied between 60 MPa and 100MPa. The time of the consolidation phenomenon which happens at the stage is very short and the amplitude of this phenomenon is difficult to control.

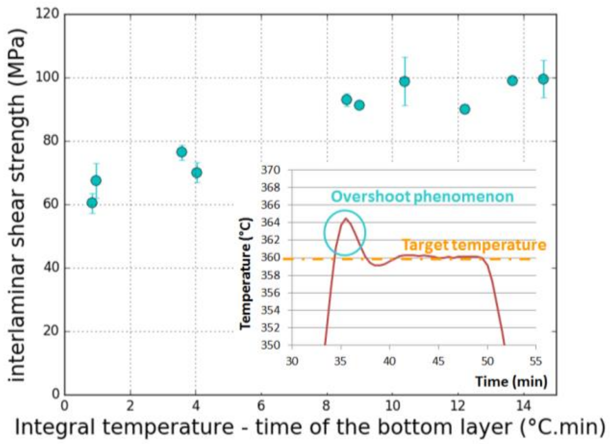

Figure 3. Influence of the overshoot phenomenon on the interlaminar consolidation. Inset enlargement of the temperature profile exhibiting the overshoot

3. Monitoring system for out-of-autoclave consolidation

3.1. Experimental set-up

We describe an experimental system to monitor the consolidation quality of 16-UD-layers laminates of carbon fiber/PEKK prepregs. The laminates were heated by an aluminum mold beneath the prepreg stack. As mentioned previously, the laminate was placed under a vacuum bag and was compacted by a pressure differential. The pressure and thermal cycles were the same as presented in Fig.1. To measure the temperature field, thermocouples of type K were placed on the top and on the bottom of the laminate. By measuring the temperature gradient between the bottom and top layers, we could characterize the thermal conductivities of the laminate and of interlaminar zones. The interlaminar thermal conductivity can be a measure of the intimate contact between adjacent layers [11, 12]. A couple of optical cameras was used to observe the evolution of the laminate's thickness during the consolidation. The data was acquired by VicSnap 8 and treated by Vic 3D.

3.2. Results and discussion

The results obtained by this monitoring system were reproducible and showed a strong correlation between the temperature evolution and the laminate thickness (cf. Fig. 4). Two distinguished peaks of temperature gradient are observable during the consolidation cycle: the one at the Tg and the other at the Tm. The evolution of the thickness until 300°C could be explained by thermal expansion of the matrix in the transverse direction, which was in agreement with independent thermal expansion test results. The reduction of the thickness at the melting temperature can be due to the flattening of asperities at the prepreg layer surface.

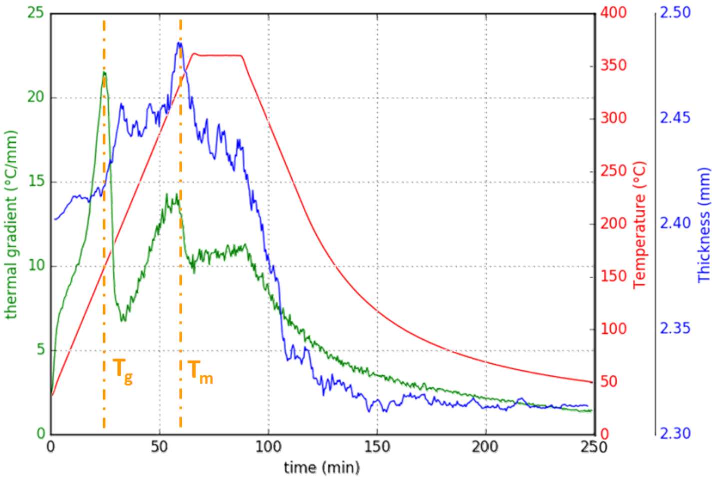

Figure 4. Result of consolidation process monitoring: the thermal gradient in the laminate, the mold temperature and the laminate's thickness during the consolidation cycle

The sudden decrease of the thermal gradient at the Tg corresponds to a diminution of the thermal contact resistance and to an establishment of intimate contact between layers. This intimate contact is established by virtue of the movement of the molecule chains which are in amorphous parts. We also investigated the quality of laminate consolidation for different initial crystallinity degrees (see Fig.5). From the results, we could observe that for the laminate fabricated from the highly crystallized prepregs (around 30%) there was no decrease of the thermal gradient at the Tg (see Fig.5). Thus, molecule chains in crystallized matrix did not take part in the interlaminar contact establishment. The required time to establish the contact between the layers was greatly increased because of the small quantity of molecule chains which took part in this phenomenon. Nevertheless the thermal contact resistance at the interlaminar zones was decreased at the Tm in all the cases. Therefore, a good consolidation quality was finally obtained as shown by the ILSS results (see the small graph in Fig. 5).

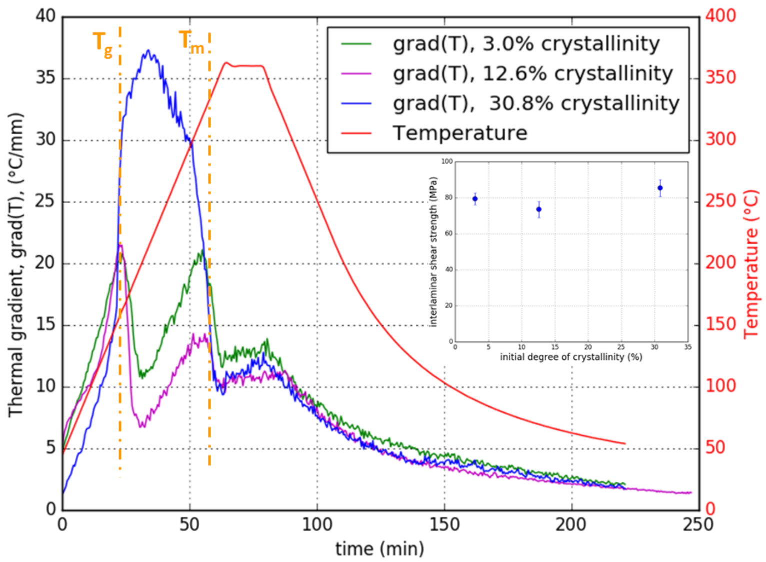

Figure 5. Influence of the initial degree of crystallinity on the consolidation phenomenon at the Tg.

3.3. Modelling of the heat transfer

The heat transfer analysis was performed by finite element method using Comsol© (see. Fig.6). The system to analyze was a laminate with 16 carbon/PEKK prepreg layers and 15 interlaminar zones whose thickness was 10μm. This value was around twice the size of the asperities on the prepreg surface. We considered that the thermal conductivity of these interlaminar zones varied, at Tg, from a very low conductivity under the vacuum condition to that of the matrix. The laminate was submitted to a heating on the bottom side and to a convection on the top side. The boundary conditions were adiabatic at both sides. From the results, we observed that the peak of the thermal gradient at the Tg was correctly predicted by the model. Thus, we can conclude that the peak of the thermal gradient at Tg is an effective index to monitor the evolution of interlaminar thermal conductivity and the establishment of interlaminar contact.

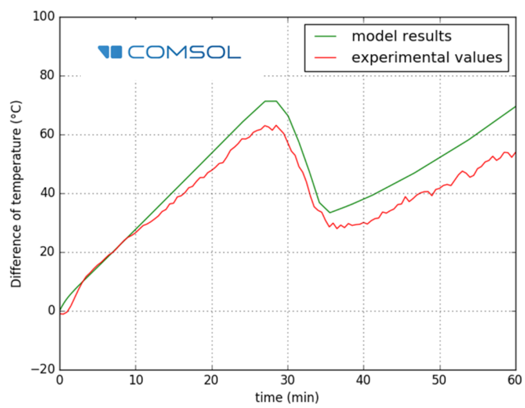

Figure 6. Comparison of the temperature gradient between experimental and simulation results.

4. Conclusions

By out-of-autoclave consolidation applying low pressure, we can manufacture thermoplastic composites with high interlaminar shear strength. The manufacturing conditions should be carefully optimized, however. In particular, the compaction pressure has a high impact on the consolidation’s quality. A compaction pressure above 700mbar is necessary to obtain a high interlaminar shear strength. For the heating cycle, the maximum temperature of at least 360°C is required whereas there is a high sensitivity to reach this maximum temperature. Finally the cooling rate and the corresponding crystallization of the matrix have no significant influence on the quality of laminate consolidation.
Moreover we identified the major phenomena to evaluate the laminate consolidation during the manufacturing cycle: the one at the Tg and the other one at the Tm. Both can be detected by peaks of the thermal gradient through the thickness direction of laminate. In particular, more interesting is the peak of thermal gradient at the Tg which is due to the molecule chains which are initially in amorphous configuration. Thus, the more the prepreg is crystalline, the lower the thermal gradient and the slower the establishment of interlaminar contact. The interlaminar zones which are initially vacuum zones are shrunk and the matrix fills these empty zones. Consequently, the thermal conductivity of these zones is greatly increased at this step of the consolidation. A delay in the establishment of interlaminar contact does not lead to a diminution of the final mechanical properties of the laminate because of the matrix flow at the Tm which fills the interlaminar gaps and improve the intimate contact. Therefore, the matrix melting at the Tm is the key parameter to the laminate consolidation.

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