Layer by layer curing (LbL)
Feasibility study final report

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Executive summary

The feasibility study funded by the EPSRC Future Composites Manufacturing HUB entitled 'Layer by layer curing (LbL)' was carried out in the period from 20/11/2017 to 01/06/2018. The study aimed to test if the LbL process is feasible by carrying-out computer simulation of LbL curing, designing the process to achieve an adequate compromise of interlaminar properties and efficiency, implementing physically the whole layer variant of the process and assessing the quality of laminates produced.

The investigation was carried out using a highly exothermic prepreg system (Hexcel 913/glass). The cure and consolidation behaviour of the prepreg were characterised and captured in appropriate constitutive models and the coupled simulation was executed for scenarios of the whole layer and ATL variants of the process. The simulation was used to optimise the curing of a 40 mm thick laminate for both the LbL process and conventional processing subject to the same temperature overshoot constraints to allow designing the LbL process conditions and comparing against conventional processing. The cure using the LbL process can be achieved within about 1 h 20 min in comparison to about 3 h for the optimised conventional cure process. This significant benefit is accomplished within the actual stage of deposition of the material, which assuming a consolidation stage duration similar to curing means that the overall LbL process can be completed in less than one quarter of the duration of the conventional process. Furthermore with the LbL process, there are profiles that can complete the cure of the 40 mm thick laminate with moderate or low temperature overshoot. The LbL process can exploit process time savings found by eliminating thermal mass heat up/down of tooling by processing at isothermal conditions. This is not possible with conventional curing at this level of thickness with reactive thermosetting polymers. The process was implemented using a servo-hydraulic machine equipped with heated plates. The setup produced specimens of 40 mm thickness with acceptable temperature overshoot during the process in agreement with the simulation. Furthermore, the temperature distribution evolution measured during the process and the thickness of the consolidated part matched those predicted by the simulation proving the validity of the modelling analysis and optimisation results. The quality of the LbL laminates was investigated using microscopy and was found to be adequate with porosity kept at low levels, microstructure/morphology similar and finer than in conventional processing. The mechanical behaviour of material produced using the LbL route was compared with that of conventional laminates. Interlaminar shear strength (ILSS) and double cantilever beam (DCB) mode I toughness testing showed that laminates produced using LbL curing match those of conventional laminates.

Overall the feasibility study delivered a 75% reduction in process time without degrading product quality by achieving equivalent interlaminar properties and porosity.

1. Introduction

This report summarises the outcomes of the feasibility study entitled 'Layer by Layer curing (LbL) funded by the EPSRC Future Composites Manufacturing Hub and carried out in the period from 20/11/2017 to 01/06/2018. The main concept tested in the study was the merging of the curing stage with consolidation and deposition through the elevated temperature processing of sublaminates placed sequentially to build the produced component. The main challenge addressed was the management of thermal profiles to achieve an optimum compromise between accelerating the process and controlling the partial cure of layers already placed on the stack so that sufficient adherence of subsequent sublaminates could be achieved. This was addressed using simulation of the consolidation and cure and demonstrated experimentally in process trials carried out with a prototype setup developed in the project. Furthermore, the quality of LbL process product was assessed using microscopy and mechanical testing.

This report is organised in five sections. The second section sets the outcomes of the study against planned objectives and deliverables as well as the overall aim and targets. The third section summarises the research activity and its technical outputs in the form of a draft journal paper. The fourth section outlines the Hub core proposal planned aiming to establish the LbL process at the scale and level of complexity required for advanced applications to composite structures. The fifth contains a poster presentation summarising the outcomes of the feasibility study.

2. Feasibility study outcomes versus aim, objectives, deliverables and targets set

The overall aim of the study was *to establish the capability of producing composites by processing in a single layer by layer (LbL) step*. This was achieved by implementing successfully the whole layer by layer process driven by the results of process simulation and optimisation and assessing the quality and performance of the produced laminates. Delivery with respect to the specific objectives set is as follows:

O1. Simulation of the layer by layer process - including consolidation, thermal and curing effects - to enable investigation of process scenarios to be carried out. Cure and consolidation constitutive models were developed based on a testing campaign and implemented within existing 1D simulation tools. The tools were coupled and use to optimise the process. Results obtained during process trials allowed successful validation of the model.

O2. Evaluation of interlaminar properties at interfaces produced using partially cured sub-laminates to establish material state limits appropriate for adhesion of successive layers. A characterisation campaign carried out using specimens produced by consolidating and co-curing partially cured sublaminates with fully uncured sublaminates. Interlaminar shear strength (ILSS) and double cantilever beam (DCB) tests were carried out and showed that mechanical performance of the interface is preserved up to the gelation conversion of the epoxy matrix at about 60% of partial curing

O3. Process optimisation to identify conditions combining high speed and sufficient layer adhesion. Simulation was carried out to identify thermal profiles for the LbL process that deliver a degree of cure sufficient for layer adhesion whilst producing an acceptable level of temperature overshoot and minimising process time. Optimised profiles were identified and subsequently implemented in process trials. Furthermore, the simulation was utilised to optimise conventional curing by minimising process time subject to an overshoot constraint to allow evaluation of the efficiency of LbL curing.

O4. Implementation and demonstration of the whole layer variant of the process. The process was implemented by adapting a hydraulic press. Curing trials of a thick (40 mm) laminate were carried out under the conditions identified by simulation and optimisation. Thermal monitoring was also incorporated in the process to allow quantification of thermal overshoots and comparison with model results for validation purposes.

O5. Assessment of product quality to validate the development. Material quality was assessed using microscopy and through thickness bending testing. Electron and optical microscopy showed that the levels of porosity present in the LbL produced material are negligible and do not exceed those of conventionally produce laminates. The microstructure of the LbL produced materials is finer than that of conventionally produced laminates. Though thickness bending tests indicated a strength that exceeds that measured for in plane transverse laminates.

Full completion of project deliverables was achieved as follows:

D1. The definition of the process envelope of time and distribution of cure state of the LbL process for scenarios of whole layer or local placement and conventional or fast curing systems. The combination of targets set by the mechanical characterisation of partially cure interfaces with simulation developed were utilised to optimise thermal profiles. Scenarios of low and moderate to high temperature overshoots were addressed. Processing profiles for Automated Tape Laying (ATL) were also investigated for components of varying thickness and in-plane dimensions.

D2. The physical implementation of the whole layer by layer variant of the process. The process was implemented successfully and instrumented process trials were carried out. Thick laminates were produced successfully within the times predicted by the simulation.

D3. Validation of product quality for the whole LbL variant. Assessment using microscopy and mechanical testing showed the materials produced using LbL curing has quality equivalent to that of conventionally produced material.

The target of the feasibility study was a *reduction of total process time by more than 75% at product quality equivalent to that of conventional processing*. This was achieved for the thick laminates produced in process trials and established for other geometries through simulation.

3. Overview of the research – Journal paper in preparation entitled 'Layer by layer manufacturing of composites'

The work presented in this paper puts forward a new manufacturing strategy for the processing of thermosetting composites based on Layer by Layer (LbL) curing. The process operates additively. Sublaminates or individual layers are placed in a heated press, they are partially cured while consolidating and the press is raised to load the next layer or sublaminate following the same cycle until completion of the part. Coupled consolidation-cure simulation was utilised to design the process and establish its capabilities and efficiency benefits. Mechanical testing was carried out to assess the interfacial properties of material produced by the LbL route. Simulation showed that benefits in terms of cure time correspond to halving its duration while merging it with consolidation resulting in a much higher overall process duration improvement. Furthermore, the LbL process can access areas of the process design space that are inaccessible with conventional processing, with its benefits intensified as the thickness of components increases. Mechanical testing showed that for pre-cure of placed layers below the degree of cure at which gelation of the matrix occur, the interlaminar properties of material produced using the LbL process is equivalent to that of conventionally manufactured material. A process trial was carried out demonstrating successfully the LbL manufacturing route. On line measurement of temperature and compaction matched the prediction of the simulation, whilst the quality of the material produced was found to be equivalent to that of composites produced using standard processing routes.

3.1. Introduction

Placement, consolidation, and curing have historically remained separate and sequential steps carried out to manufacture composites structures. Carrying out placement and consolidation at relatively low temperatures to avoid curing implies slower material response and longer time scales required for consolidation. Furthermore, curing the material in bulk costs more in time as thermal lags in the low thermal conductivity thickness direction need to be equilibrated. The inefficiencies of the current processing philosophy are exaggerated as component dimensions - especially thickness - increase. Placement/forming and consolidation often need to be carried out in stages, while thermal lags increase non-linearly with thickness. For the largest structures, there is a significant challenge to lay up the part inside the out-life of the prepreg. In the case of thick and ultra-thick components, thermal profiles need to be conservative and long to eliminate detrimental temperature overshoots [1].

Efforts to combine processes into a single Layer-by-Layer (LbL) have been investigated for thermoplastic composite materials. The main obstacle is still matching the interlaminar properties achieved through LbL to those when a subsequent autoclave consolidation step is used [2]. In this regard, lower viscosity thermosetting materials may allow combining all three steps if final fibre volume fraction is retained, while advancing the degree-of-cure to the appropriate level where adhesion is retained across subsequent ply interfaces. White and Kim [3] proposed a curing technique to process thick composites by stages without exothermic overshoots; sublaminate stacks were consolidated individually at low temperature to build up thickness, followed by a final cure. Staged curing advanced the degree-of-cure to a point where both temperature overshoot was reduced and interlaminar properties were retained because the polymer remains only partially cured until the final cure stage. As a result, adhesion and consolidation are possible with this approach, but the total process time was much longer than a conventional curing cycle.

An LbL process aims to provide sufficient consolidation quality in each placement stage. Experimental definition of conditions where placement, consolidation, and cure are delivered without compromising adhesion might be possible. Numerical modelling tools are sufficiently well advanced and capable of defining the optimal conditions. Cure and consolidation models are

available in the literature [4-8], alongside appropriate methods for material property characterisation [6, 9, 10].

The work presented here addresses the main developments for demonstrating the feasibility of an LbL process for thermosetting composites. The process concept is defined and simulation tools are adapted to model the coupled cure-consolidation behaviour under one-dimensional conditions. The mechanical behaviour of interfaces produced form material with different states of pre-cure is established and an instrumented process trial of the LbL process is carried out using a setup adapted for this purpose.

3.2. Process concept

The concept proposed in this work addresses the limitations and inefficiencies of current strategies through layer by layer cure of prepreg during placement/consolidation. The process takes place in distinct steps of consolidation and partial curing on a heated tool, with the upper layer heated through contact in a press. The process is illustrated schematically in Fig. 1. The process starts with a layer or sublaminate placed on the heated lower tool. The upper tool, which is also heated, is brought in contact and held in place to consolidate the material until the cure has progressed sufficiently. Subsequently, the tool is raised and the next layer or sublaminate is placed on top of the partially cured material. Press application follows, to achieve consolidation and cure of the new layer or sublaminate. This sequence is repeated until the whole of the thickness in built up. This processing strategy results in periodic heating and compaction of the layers already placed in the stack.

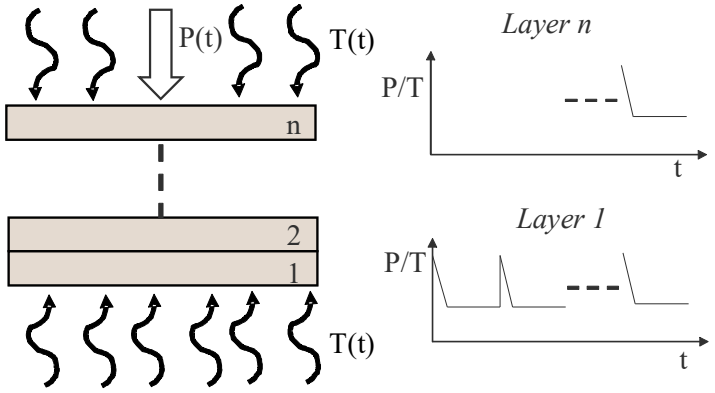


Fig. 1 LbL process concept

The main advantage of the LbL processing strategy is that it allows fast processing of a fraction of the thickness of the laminate. The fact that the process operates in a fraction of the thickness permits use of aggressive thermal profiles. This can result in significant acceleration of the overall process. Consolidation under higher than usual temperature can achieve faster compaction and void removal for the relatively thin layer of material in contact with the top tool. Similarly, the low thickness of the active layer or sublaminate allows acceleration of the cure as heat transfer is faster and heat dissipation more effective when required to ameliorate the effects of exothermicity of the curing reaction. The latter is of paramount importance in thick and ultrathick laminates, where the potential of uncontrollable temperature overshoots makes necessary the use of highly conservative and long cure profiles.

Successful implementation of the proposed concept involves challenges in controlling the cure process to achieve sufficient progress without compromising adhesion of adjacent layers and in the selection of processing rates ensuring adequate consolidation quality and void removal. The former can be achieved by investigating the mechanical behaviour of interfaces produced between partially

pre-cured material and fully uncured material upon cure of the whole laminate to identify the level of partial pre-cure that allows sufficient adherence and interfacial strength. The latter can be accomplished by exploring the interplay between acceleration of the cure reaction and acceleration of the consolidation process with increasing temperature, with the aim of establishing processing conditions that allow a high level of prepreg compaction before cure progresses sufficiently to increase the viscosity resin beyond processable levels. The processing characteristics of high performance epoxy prepreg, which are based on the autocatalytic nature of epoxy cure, lend themselves to this type of strategy.

3.3. Methodology

3.3.1. Materials and manufacturing

Composite specimens were manufactured using glass reinforced 913 epoxy matrix unidirectional (UD) prepreg supplied by Hexcel (913G-E-5-30%). The prepreg contains 30 wt.% resin and has a fibre areal density of 192 g/m^2 corresponding to a cured thickness of 125 μm for a fibre volume fraction of approximately 60%. HexPly® 913 is a modified epoxy with a density of 1.23 g/cm^3. The fibres are E-glass with a diameter of 5 μm. For thin laminates, below 3 mm, the matrix systems can be cured in a single dwell in the 125-160 °C range. For greater thicknesses the exothermicity of the resin system necessitates the utilisation of a low temperature dwell, below 100 °C, before the final dwell.

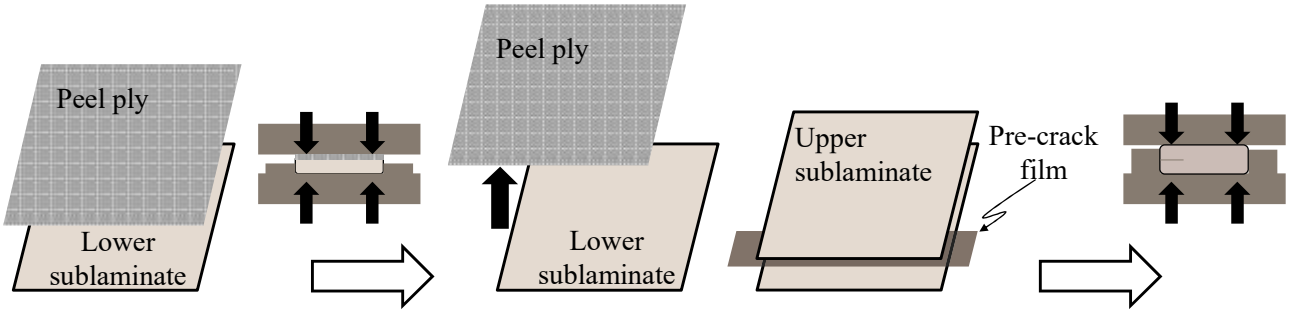
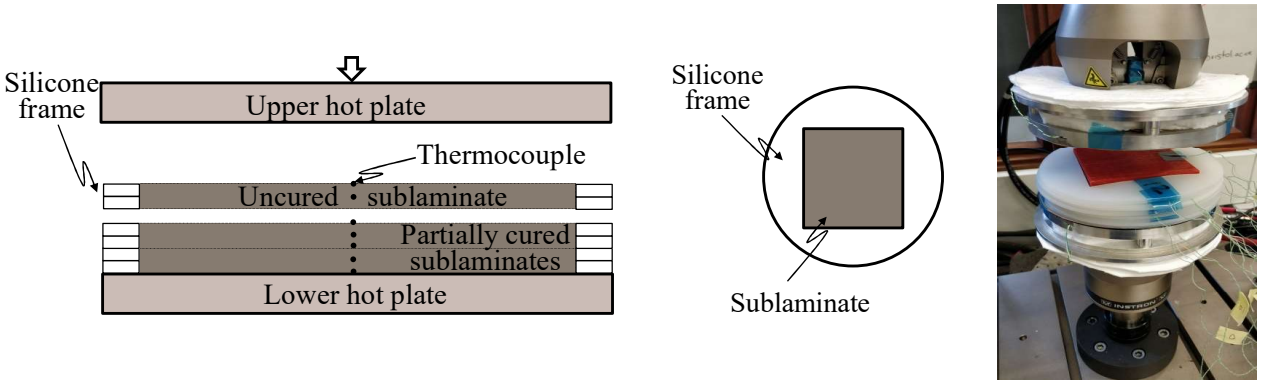


Fig. 2 Procedure used for the production of partially pre-cured interface specimens

Composite plates with an interface at mid-plane comprising one side of partially pre-cured material at prescribed levels and one side with fully uncured material prior to final cure were manufactured using a Fontijne Grotnes LabPro 1000 two column press. The plates were used to characterise the influence on interfacial mechanical properties of partially pre-cured material on one side before the final step of processing. The procedure, which is illustrated schematically in Fig. 2, comprised three steps. Initially, two halves of 15 layers of UD prepreg with 300 × 300 mm in-plane dimensions were laid up. One half was placed in the press between PTFE coated steel plates and a fixed height spacer with a thickness of 2.15 mm and cured under 20 kN following a thermal profile appropriate for yielding the prescribed level of pre-cure. The top surface of the laminate was covered with a layer of nylon peel ply (PeelPlyN85 VAC Innovation Ltd.) aiming at generating a controlled level of roughness on the pre-cured surface. A PTFE coated thermocouple (k-type) was placed in the centred of the assembly in contact with the steel platen to allow measurement of the actual cure profile and subsequent calculation of the degree of pre-cure. After completion of curing and cooling down, the peel ply was removed and the uncured half was placed on top of the pre-cured sublaminate. An 80 mm wide strip of 10 μm thick FEP crack starting film was inserted between the two halves aligned to one edged of the plate. The stack was placed in the press between the two PTFE coated plates adding a 2.35 fixed height spacer to reach a total thickness of 4.5. Curing was

carried out under a pressure of 20 kN using a ramp rate of 2 °C/min to 100 °C, a 10 min isothermal dwell followed by a 2 °C/min ramp to 160 °C and a second 10 min dwell. The plates were cut to the appropriate specimen dimensions for subsequent testing using a diamond impregnated saw blade (Crandon 60/85).



(a) (b) (c)

Fig. 3 LbL process setup: (a) section of setup; (b) top view of silicone frame with sublaminate; (c) experimental setup

A 40 mm thick 913 epoxy/glass laminate was manufactured using the LbL process. The manufacturing was carried out using an Instron servo-hydraulic 8801 universal testing machine equipped with two temperature controlled heated plates clamped by the grips of the testing machine. The process and geometry are illustrated schematically in Figs. 3.a and 3.b. and the setup is shown in Fig. 3.c. Three hundred 100 × 100 mm prepreg sheets where cut and laid up to produce 6 sublaminates each following a $[0/90]_{25}$ layup sequence. A k-type thermocouple was placed at the mid-plane of each sublaminate as well as on its top surface. Circular 2.5 mm thick silicone sheets with a diameter of 180 mm and a 102 mm square centre cut-out (Fig. 3.b) were prepared to constraint the sublaminates and to prevent excessive resin bleeding from the prepreg during compaction. Each sublaminate was positioned in the cavity formed by two silicone sheets. During the process both heated plates were kept at 130 °C. The first sublaminate was placed on the lower heated plate and a compressive force of 1 kN was applied at a rate of 100 N/s. After 10 min the upper plate was raised and the next sublaminate was placed in the assembly. The duration of the material loading step, during which the underlying sublaminate, was cooling down was 1-2 min. This sequence was repeated 6 times to produce the 40 mm thick laminate. A diamond impregnated saw blade was used to cut the laminate, followed by grinding using 1200 and 2400 grit silicon carbide paper and polishing with 9 μm diamond spray to prepare microscopy samples.

3.3.2. Simulation

The compaction and cure of the material were simulated using an 1-D model. The flow-compaction model is based on [5] and has the capability to capture both squeezing and bleeding/percolation flow, which makes it an ideal solution for modified thermosets, such as the epoxy resin used in this study. Consolidation stress (σ) is linked to the strain rate in the thickness direction ($\dot{\varepsilon}$) as follows:

$$\sigma = \eta_{strain}(\varepsilon)\eta_{rate}(\dot{\varepsilon})\dot{\varepsilon} \quad (1)$$

where η_{strain} and η_{rate} represent the elastic and viscous behaviour of the overall apparent viscosity of the prepreg.

The viscous term follows a power law:

$$\eta_{rate}(\dot{\varepsilon}) = e^{\bar{b}}(-\dot{\varepsilon})^{a} \quad (2)$$

where a and $\bar{b}$ are resin material and temperature dependent parameters estimated experimentally.

The elastic term incorporates the transition between squeezing and bleeding flow that occurs when shear deformation at the edges of the prepreg reaches a critical locking strain (ε^l). The strain dependent term in Eq. 1 is decomposed into a term expressing effects at ply level (η_{ply}) and a term representing microscale phenomena (η_{mirco}):

$$\eta_{strain}(\varepsilon) = \eta_{mirco}(\varepsilon)\,\eta_{ply}(\varepsilon) \tag{3}$$

The ply term depends on the size of the prepreg layer and is expressed as follows:

$$\begin{aligned} \eta_{ply} &= 2\left(\frac{w_0}{h_0}\right)^2 e^{-4\varepsilon}, \quad \varepsilon < \varepsilon^l \\ \eta_{ply} &= 2\left(\frac{w_0}{h_0}\right)^2 e^{-2(\varepsilon+\varepsilon^l)}, \quad \varepsilon \geq \varepsilon^l \end{aligned} \tag{4}$$

Here w_0 and h_0 are the initial width and thickness of the ply respectively, whilst the locking strain (ε^l) is computed based on geometrical considerations [5].

The microscale term is:

$$\begin{aligned} \eta_{micro} &= 2\eta_{resin}\sqrt{\chi_l}e^{\varepsilon}k\left[\left(\frac{k}{\sqrt{\chi_f}e^{\varepsilon}-k}\right)^2+3\right], \quad \varepsilon < \varepsilon^l \\ \eta_{micro} &= 2\eta_{resin}\left(\frac{l_0}{d}\right)^2 k\left(\frac{k}{\sqrt{\chi_f}e^{\varepsilon}-k}\right)^2, \quad \varepsilon \geq \varepsilon^l \end{aligned} \tag{5}$$

where l_0 is the fibre length, d is the edge length of the fibres in the direction perpendicular to their orientation, k is a phenomenological parameter estimated experimentally, η_{resin} is the resin viscosity and χ_l and χ_f are the aspect ratios of a unit cell at locking and at the compaction limit respectively. The aspect ratio at locking can be computed directly from the true locking strain as:

$$\chi_l = e^{-2\varepsilon^l} \tag{6}$$

The aspect ratio at the compaction limit can be approximated as $\chi_f \approx 1.4\chi_l$ [5].

The ordinary differential equation system expressed by Eqs. (1)-(6) is solved using MATLAB ODE® Solver15s. The three empirical parameters of the model ($a, \bar{b}, k$) are estimated using the result of compaction experiments and curve fitting.

The cure solution is based on the 1-D energy balance with heat conduction and generation terms:

$$\rho c_p \frac{\partial}{\partial x} = \frac{\partial}{\partial x} K \frac{\partial T}{\partial x} + \left(1-\upsilon_f\right)\rho_r H_{tot}\frac{d\alpha}{dt} \tag{7}$$

Here K is the thermal conductivity of the composite in the through thickness direction, ρ and $c_p$the density and specific heat capacity of the composite respectively, υ_f the fibre volume fraction, ρ_r the density of the resin, H_{tot} the total enthalpy of the curing reaction, whilst α denotes the degree of cure of the resin, T the temperature, t the time and x the spatial coordinate in the thickness direction.

The heat generation term is based on the assumption that heat release rate is proportional to the rate of reaction ($d\alpha/dt$). The rate of reaction is expressed as a function of the instantaneous temperature and degree of cure, with the following expression selected for the 913 epoxy resin system:

$$\frac{d\alpha}{dt} = k_1(1-\alpha)^{n_1} + k_2(1-\alpha)^{n_2}\alpha^m \tag{8}$$

Exponents n_1, n_2 and m are phenomenological parameters corresponding to reaction orders and k_1, k_2 are rate constants following an Arrhenius dependence on temperature:

$$k_i = \frac{A_i e^{\frac{E_i}{RT}}}{1+e^{C[\alpha-\alpha_T(T-273.15)-\alpha_c]}}, \quad i = 1,2 \tag{9}$$

where E_1 and E_2 are activation energies, A_1 and A_2 are pre-exponential factors, R is the universal gas constant. The denominator in Eq. (9) incorporates diffusion limitations to the cure kinetics model, which are of significance when the glass transition temperature of the curing resin approaches the curing temperature. This is carried out through a critical degree of cure value, which is a function of temperature and is controlled by phenomenological parameters α_T and α_c , whilst the breadth of the transition to diffusion controlled reaction is governed by parameter C. It should be noted that Eq. 9 uses absolute temperature.

Solution of the differential equation expressed by Eq. (7) coupled with the cure kinetics model expressed by Eqs. (8) and (9) is carried out using a finite element modelling solution of the problem implemented and validated in [4]. The thermal properties are assumed to be constant, whilst the cure kinetics parameters are estimated using differential scanning calorimetry results and curve fitting. The relevant analysis is presented in the following sections.

The two models are coupled is a weak scheme, which is illustrated in Fig. 4. The simulation starts with the first sublaminate and the execution of the heat transfer simulation of the cure. This generates a temperature distribution which is fed into the compaction simulation to compute the thickness of the sublaminate. This is fed back to the heat transfer model, which using the updated thickness, computes a new temperature distribution. This is first checked for convergence by comparing with the previous iteration and if convergence is not reached the consolidation model is executed again with the updated temperature. This procedure is repeated until convergence. Subsequently, the next sublaminate is added and the simulation starts using the temperature and degree of cure of the existing sublaminate as initial conditions of the heat transfer solution for the corresponding part of the finite element mesh. The iterations with the consolidation model are executed by using the temperature distribution since the beginning of the process and updating only the new increments corresponding to the new sublaminate. This procedure is repeated until all sublaminates are placed and the process is complete. It should be noted that in execution of the simulation for the material system of this investigation the solution converged within the second iteration as a result of the full dominance of bleeding flow resulting in very fast approach to the final thickness of the layers.

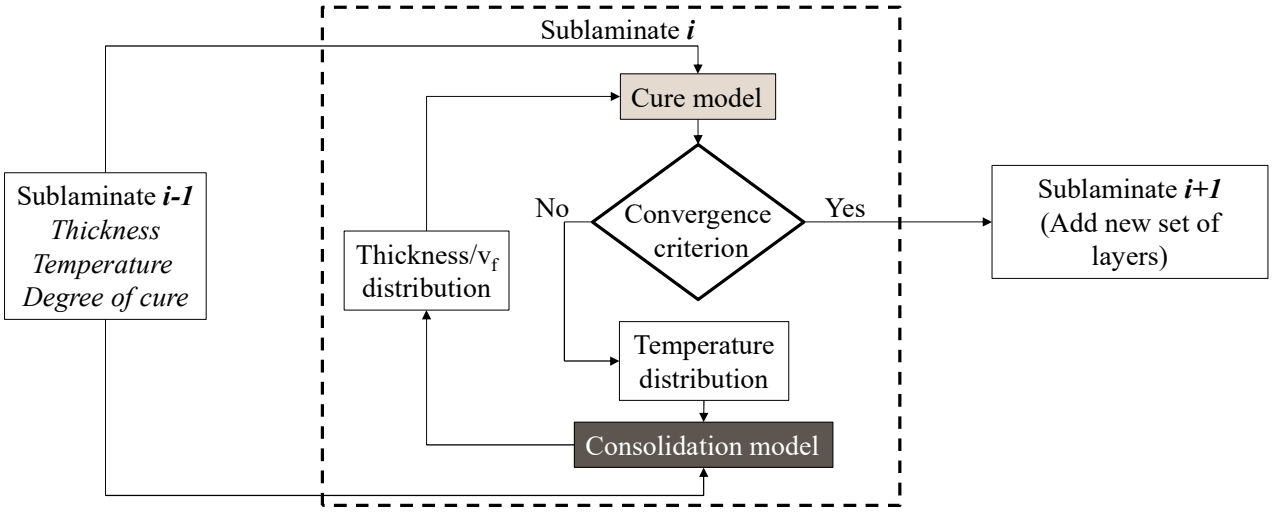


Fig. 4 Simulation flow

3.3.1. Characterisation and mechanical testing

The parameters of the consolidation model were derived from ramp-dwell compaction tests under isothermal conditions. Rectangular sheets of the prepreg, with in-plane dimensions of 25 × 50 mm, were laid up to form a crucifix geometry with a stacking sequence of $[0/90]_8$. This arrangement

results in an effective 25 × 25 mm square region in the centre of the specimen with a mean thickness of 2.4 mm. This geometry minimises lateral loss of material from the edges of the central square area as the plies are squeezed in the through-thickness direction. The tests were carried out using an Instron servo-hydraulic 8801 universal testing machine and the same heated plates and camera setup as the one utilised in LbL process trials. The tests were performed at 30, 60 and 90 °C. The compaction load was 80 N initially and was increased in 4 steps of 40 N each, with the load kept constant at each force level for 240 s. The highest load was 240 N and the overall duration of the experiment 1200 s. The loading rate was 65 N/s. In terms of pressure levels these conditions correspond to pressure levels of 0.13 to 0.38 MPa with a step of about 0.06 MPa and a loading rate of 0.1 MPa/s. Three specimens were used for each test case.

Differential scanning calorimetry (DSC) was utilised to identify the parameters of the cure kinetics model. The experiments were carried out using a TA-instruments DSC Q200 apparatus. Nitrogen purge gas with a flow rate of 50 ml/min was used. Samples of the prepreg of 4-8 mg weight were encapsulated in Tzero™ pans and lids. Isothermal experiments were carried out at 90, 110, 130 °C and dynamic tests at 1, 2, 5 and 10 °C/min. The material was cured fully after the end of isothermal experiments in a heating ramp at 10 °C/min to allow measurement of residual heat, which was subsequently used to estimate the total reaction enthalpy per sample. This is necessary in prepreg samples as the variability in resin content does not allow calculation of reaction rates based on total enthalpies measured in independent dynamic tests. The DSC data were integrated to calculate the degree of cure and reaction rate evolution using a horizontal baseline for isothermal conditions and sigmoidal for dynamic conditions. All DSC tests were duplicated. The specific heat capacity of the prepreg was measured using the DSC apparatus in modulated mode. Tests were performed at 90, 110 and 130 °C. The period and amplitude of the modulation were 1 min and 1 °C respectively. An average specific heat capacity was calculated over all tests and data points as the simulation uses a constant value for this property.

Interlaminar shear strength (ILSS) short beam tests were carried out to measure the dependence of ILSS on the degree of pre-cure of one side of the interface. The testing procedure followed British Standard BS ISO 14130. The 3-point bending tests were performed in an Instron 5500R testing machine with a 30 kN load cell using specimens of 23 mm in width, 4.5 mm in thickness and 22.5 mm in span length, resulting in a 5:1 span to thickness ratio. The loading rate was at a constant speed of 1 mm/min. Five specimens were tested for each case. After each test, the failure mode of the specimen was inspected to ensure the occurrence shear failure mode without any presence of compressive and tensile failure in specimens used for subsequent analysis.

Mode I double cantilever beam tests were carried out on an electro-mechanical Zwick Z010 test machine with a 2 kN load cell. The test procedure followed the British Standard BS ISO 15024. The specimens dimensions were 140 × 20 × 4.5 mm. Aluminium loading blocks (15.5 × 20 × 8 mm) were bonded on the specimens using Huntsman Araldite® 420 A/B. Three specimens were tested for each case. The specimens were painted and marked every millimetre on both sides in order to record the crack length. The cross-head speed was 1 mm/min. The applied load and crosshead displacement were recorded automatically and the crack length was determined visually and recorded alongside the corresponding load and displacement regularly. The initiation fracture toughness was determined using the 5%/max point. The corrected beam theory (CBT) was used to analyse the data. Correction factors were taken into account to compensate for the rotation at the delamination front due to asymmetrical specimen clamping, stiffening and rotation due to loading blocks and reduction of the lever arm at large displacements due to rotation at the end of the specimen. Average strain release rates were computed using the toughness values in propagation for crack lengths between 40 and 110 mm.

Polished cross sections of the material produced in LbL trials and conventional press curing were analysed using scanning electron microscopy (SEM, Philips, XL 30 SFEG) and optical microscopy

(Olympus BH50). Scanning Electron Microscopy samples were coated by sputter deposition (Polaron Equipment Ltd., E5100) of a thin layer of conductive gold alloy.

Data underlying this study can be accessed through the Cranfield University repository at http://dx.doi.org/******/cranfield.rd.******.

3.4. Results and discussion

3.4.1. Constitutive models

The results of compaction experiments are illustrated in Fig. 4. The response for all cases shows a very fast compaction in the first dwell at 0.13 MPa completed within about 10 s. This is indicative of dominance of bleeding flow as the squeezing part of the response takes place almost instantaneously. The compaction increases with increasing temperature. Similarly, the rate of compaction is also higher at higher temperatures. The compaction step at each dwell is reduced as the pressure increases. The fitting of the empirical model parameters of Eqs. (1)-(6) was carried out using the procedure detailed in [5] and the results are reported in Table 1 for the 30-150 °C temperature range. The rest of the parameters of the consolidation model are reported in Table 2. It can be observed that the model reproduces the experimental data very closely.

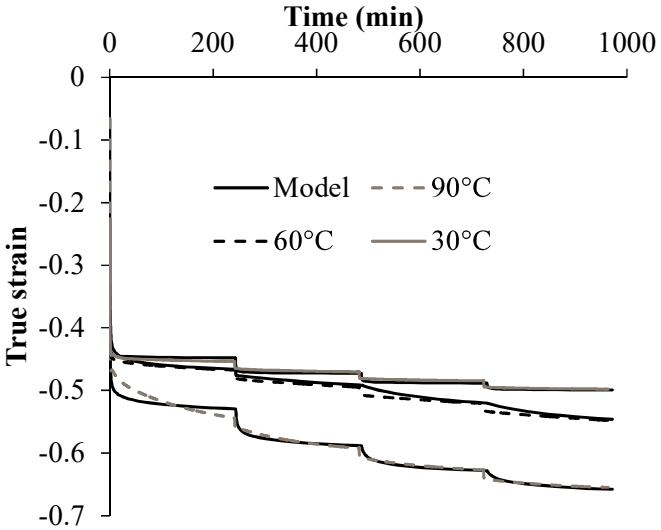


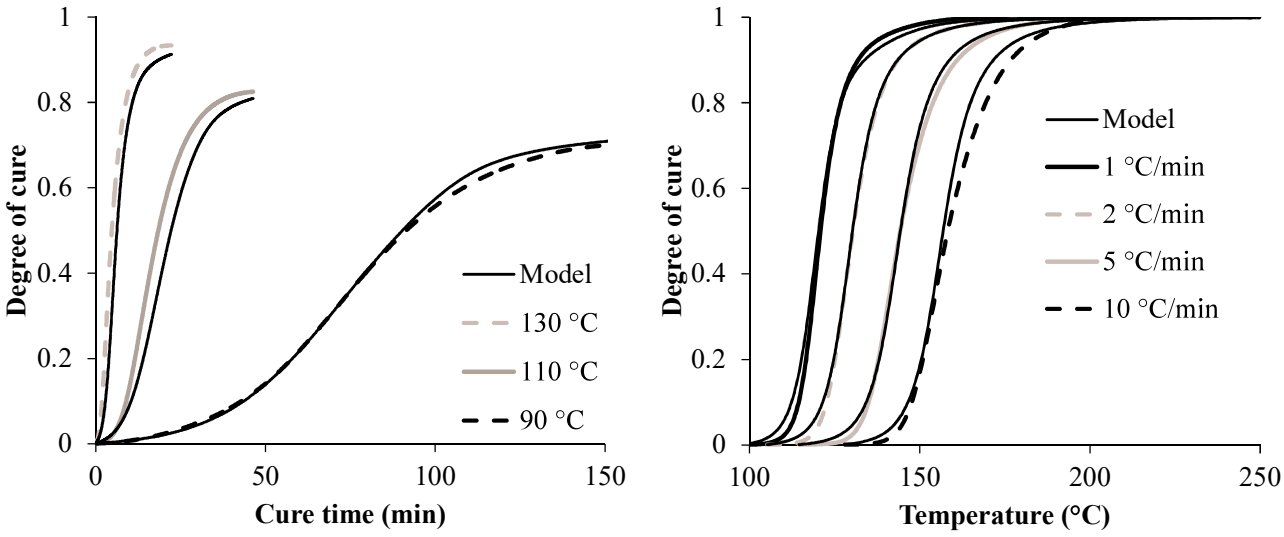
Fig. 4 Consolidation of 913/glass prepreg: evolution of compaction and model fitting for isothermal experiments

Table 1. Fitting parameters of the consolidation model over the 30-150 °C temperature range

T (°C)	30	60	90	120	150
$\boldsymbol{a}$ **(-)**	-0.92	-0.92	-0.92	-0.92	-0.92
$\overline{\boldsymbol{b}}$ **(-)**	-37.9	-36.6	-35.1	-34.4	-33.8
$\boldsymbol{k}$ **(-)**	0.79	0.68	0.62	0.58	0.54

The results of isothermal and dynamic DSC experiments are illustrated in Fig. 5. The isothermal data show the typical autocatalytic behaviour of epoxies. The reaction is accelerated by increasing temperature, whilst diffusion limitations result in partial curing under isothermal conditions. The

maximum degree of cure reached at 90 °C is around 70%, at 110 °C 80% and at 130 °C over 90%. This justifies the use of a diffusion term in the cure kinetics model expressed by Eqs. (8)-(9) to capture this behaviour. Dynamic data are consistent with the positive effect of temperature on the reaction rate. The reaction occurs at higher temperatures as the rate increases with the earlier onset being about 100 °C for the 1 °C/min experiment. The cure kinetics model using the parameters reported in Table 2 follows the experimental results very closely. The model represents successfully the temperature dependence of the evolution of cure under both isothermal and dynamic conditions. Furthermore, the model reproduces successfully diffusion limitations manifested at the latest stages of isothermal cure experiments. Table 2 also summarises the rest of the parameters of the heat transfer model.



(a) (b)

Fig. 5 Cure kinetics of 913/glass prepreg: (a) evolution of degree of cure and model fitting for isothermal experiments; (b) evolution of degree of cure and model fitting for dynamic experiments

Table 2. Parameters of constitutive models and material properties

Parameter	Value	Units
A_1	$4.46\ 10^9$	1/min
A_2	$1.67\ 10^{10}$	1/min
E_1	90350	J/mol
E_2	79520	J/mol
n_1	4.96	-
n_2	1.88	-
m	0.98	-
C	34.5	-
α_T	0.00573	1/°C
α_c	0.150	-
H_{tot}	538.7	J/g
ρ	1.55	g/cm^3
ρ_r	1.23	g/cm^3
c_p	1.4	J/g/°C
K	0.28	W/m/°C
ε^l	0.0102	-
l_0	102	mm
d	0.017	mm^2
h_0	0.18	mm
w_0	102	mm

3.4.2. Mechanical behaviour

Quantification of the degree of pre-cure of one side of the interface of mechanical testing specimens was carried out using the cure kinetics model expressed in Eqs. (8) and (9) with the parameter values reported in Table 2. The results alongside the corresponding thermal profiles are reported in Table 3. It should be noted that for all partial cure profiles the ramp rate was 2 °C/min.

Table 3. Cure profiles used for partial curing of lower half sublaminates

Dwell temperature (°C)	Dwell duration (min)	Final degree of cure
100	8	0.15
100	12	0.30
100	18	0.50
100	23	0.64
110	30	0.85
125	30	0.94

The results of mechanical testing are summarised in Fig. 6. It can be observed that both ILSS and strain energy release rate are relatively constant up to a degree of pre-cure of 60% with plateau values of about 60 MPa and 480 J/m^2 respectively. Beyond this level of pre-cure, both interfacial properties deteriorate, with a gradual reduction to about 35 MPa for ILSS and a step drop to about 130 J/m^2 for a fully pre-cured side of interface. The transition point occurs at a degree of pre-cure which coincides with the degree of cure at which gelation is expected to happen for an epoxy of this type. Therefore, it can be inferred that the critical degree of pre-cure of the interface is dictated by the formation of a three dimensional cross linking network between the pre-cured and uncured sides of the interface as well as the ability of the material to flow for pre-cure levels below the gelation region. Beyond this level, the pre-cured side of the interface is already cross linked sufficiently and allows only partial bonding with the material of the uncured side, whilst the infinite viscosity of the material inhibits mechanical keying between the two sides of the interface. Consequently, reaching a pre-cured state just below the gelation degree of cure of the resin system appears as an optimal choice to achieve minimisation of process time and maximisation of mechanical integrity of the interface.

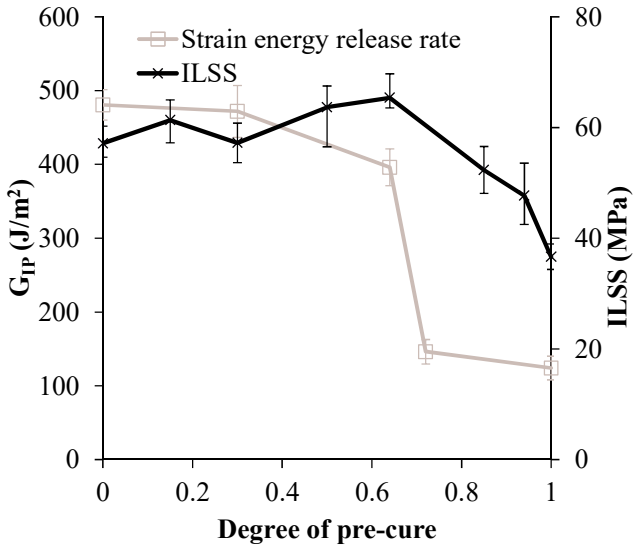


Fig. 6 Dependence of interfacial properties on degree of pre-cure of one side of the interface

3.4.3. Simulation and process design

The coupled consolidation-cure model was utilised to investigate different scenarios of the LbL process. The processing of a 40 mm thick 913/glass laminate was investigated using the models expressed by Eqs. (1)-(9) and the material parameter values reported in Tables 1 and 2. The LbL process simulated was carried out in six steps following the setup utilised for manufacturing trials. Two different cure temperatures were investigated, 110 and 130 °C, aiming at a moderate (low temperature overshoot) and an aggressive (high temperature overshoot) cure profile respectively. The duration of the dwell was determined after using the simulation to identify the duration of the heating step that would be sufficient to achieve full cure by the end of the six sublaminate cycle. A duration of 10 min was chosen for the 130 °C case and 15 min for the 110 °C case. It should be noted that according to the simulation the high temperature case results in degree of pre-cure higher then the critical conversion; however, the choice was made to allow additional pre-cure to proceed so that excessive overshoot (over 50 °C) did not occur in subsequent steps. The cooling/material loading step was set to 2 min in accordance with experimental trials of the LbL process.

The results of the coupled simulation for the two cases are illustrated in Fig. 7. It can be observed that in both cases the temperature follows a periodic behaviour. In the cycle at which a sublaminate is loaded its temperature is maximised, reaching overshoots of approximately 50 °C and 15 °C for the aggressive and moderate cure profile cases respectively. The temperature in the sublaminate goes through subsequent maxima in the following steps. These correspond to lower overshoots (around 20 °C and 10 °C for the aggressive and moderate cure profile cases respectively) in the second cycle after deposition of sublaminate, and die out after 3 cycles. This behaviour is explained by the exothermic character of the curing reaction and its diminishing role as the reaction progress through the different cycles. The maximisation of temperature occurs with a time lag in sublaminates that are already placed. The lag for the sublaminate immediately under the one deposited is about 2 °C and 1.5 °C for the aggressive and moderate profiles respectively, with this lag increasing for lower sublaminates. This is accompanied by broadening of the peak of temperature overshoot for sublaminates that are already placed. This behaviour is caused by the slow heat conduction through the thickness of the curing composite, whilst the greater lag for the aggressive scenario can be explained by the greater value of the peak temperature. The degree of cure evolution curves show that in both cases the cure of a sublaminate is complete after completion of two placement/consolidation-cure cycles. The degree of pre-cure upon placement of a sublaminate for the aggressive scenario is about 0.75 for the first placement and about 0.85 for subsequent layers. In the case of the moderate scenario, the degree of pre-cure is about 0.6 for the first layer and 0.65 for subsequent layers. The overall cure, defined as the time at which all of the laminate reaches 90%, is 70 min for the aggressive scenario and 135 min for the moderate curing scenario. The consolidation results (Figs. 7.a and 7.b) show that full compaction is achieved within a very short time within each step. The time needed is less than 1 min, which is within the time limit before the onset of the curing reaction. The level of thickness reached (about 6 mm for each sublaminate) corresponds to full compaction, which implies that the process under both scenarios shown here should achieve the level of fibre volume fraction and consolidation quality required.

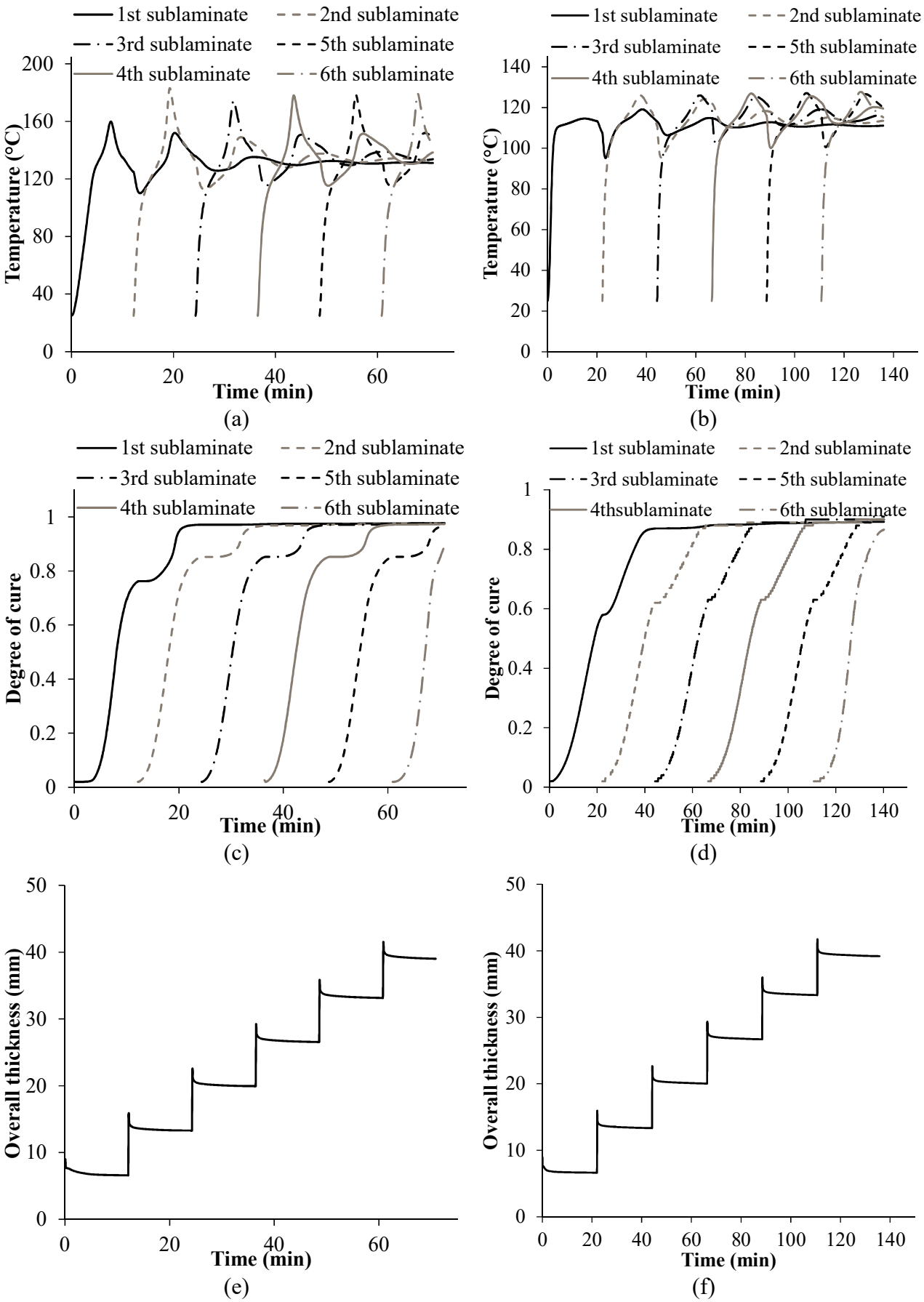


Fig. 7 Simulation of LbL process: (a), (b) temperature at mid-thickness of sublaminates; (c), (d) degree of cure on top layer of sublaminates; (e), (f) thickness evolution

Assessment of the efficiency of the LbL process was carried by comparing its performance with that of conventional processing. As conventional cure profiles tend to be conservative, the comparison was carried out against optimised profiles for the cure of 913 epoxy/glass laminates. This was carried out by evaluating the temperature overshoots and cure duration using the model described by Eqs. (7)-(9) for a 40 mm laminate cured conventionally in a single step by direct contact heating on both of its external surfaces. This is the most efficient scenario in the context of conventional processing. The cure profile was considered to comprise two dwells and heating ramps set at a constant rate (2 °C). The first dwell duration was in the 70-95 °C range, the second dwell duration in the 95-135 °C range and the duration of the dwell in the 60-240 min range. The duration of the second dwell was considered to be equal to the time required for reaching a minimum degree of cure of 90% through the whole thickness of the laminate. An exhaustive search was carried out using a step of 2.5 °C for the first dwell temperature, 5 °C for the second dwell temperature and 15 min for the first dwell duration resulting in 891 conventional process designs. The results are illustrated in Fig. 8 alongside the outcomes of the aggressive and moderate LbL process scenarios. The set of feasible solutions illustrated is limited in the range of temperature overshoot below 120 °C and of fist dwell duration below 250 min. A set of efficient solutions (Pareto set) can be identified. These solutions have the property that no improvements with respect to both objectives can be achieved by another solution in the set.

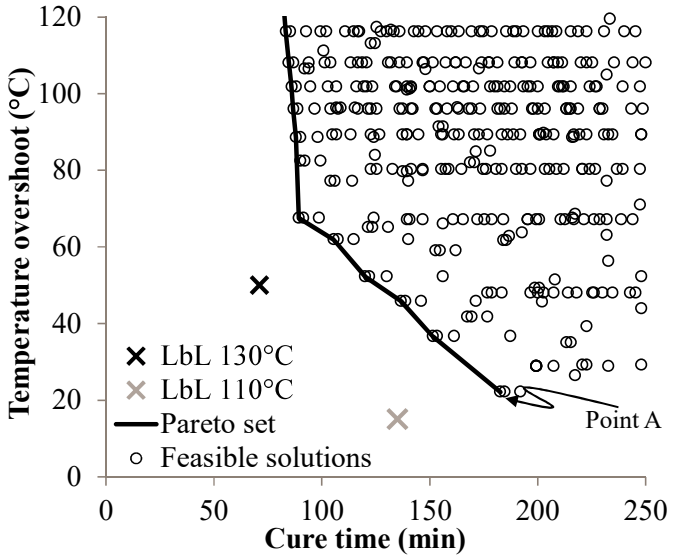


Fig. 8 Overshoot-cure duration feasible set using conventional cure of 40 mm glass/epoxy prepreg and comparison with LbL process

Comparison of the LbL process performance with conventional solutions in the Pareto set provides the most conservative assessment of the efficiency of the new process. Both LbL scenarios examined result in significant improvement with respect to the two objectives compared to efficient conventional process designs. An overshoot similar to the one occurring in the aggressive LbL process can only be achieved with a process duration greater than about 130 min using conventional curing. This compared to the 70 min required for completion of the LbL process represents an improvement of about 50%. Similarly, a process duration similar to the one achieved using the moderate LbL scenario can only be accomplished with an overshoot of about 45 °C for the conventional process. This represents a reduction of overshoot of about 70% with the LbL process. Most importantly, examination of the set of feasible solutions reveals that a cure time of 70 min, such as the one achieved with the aggressive LbL profile, is not feasible with conventional

processing. Similarly, a temperature overshoot of 15 °C, such as the one occurring in the moderate LbL process design, is not feasible with the conventional process. These results show that the conventional process has some hard limits in terms of what can be achieved for the minimisation of cure time or temperature overshoot which for the 40 mm 913/glass laminate considered here are about 90 min and 20 °C. Improvements beyond these limits cannot be achieved irrespective of how large the value of the other objective is. In contrast, the LbL process can operate in areas of the objective space inaccessible by conventional processing, allowing processing of thick laminates under conditions not feasible otherwise. Furthermore, the additive character of the LbL process means that increasing the thickness of the laminate by increasing the number of sublaminates results in the same temperature overshoot and a process duration increasing linearly. This is in contrast to conventional processing in which both process time and overshoot increase non-linearly, making the set of feasible solution smaller and eventually rendering processing beyond a thickness impossible. This limitation is lifted completely by the LbL process.

Further insight on how the conventional process achieves the cure of the laminate in contrast to the LbL process and why the latter has inherent efficiency benefits can be gained by examining the evolution of temperature and degree of cure as predicted by simulation. The solution illustrated in Fig. 9 corresponds to the process design with the lowest overshoot feasible using conventional processing (point A in Fig. 8). The specific process design involves a 150 min 75 °C first dwell and a 135 °C second dwell. The role of the first dwell is to progress the reaction as much as possible before an overshoot occurs. Once this point has been reached, the temperature is raised to carry out the rest of the reaction during the dynamic ramp and the second dwell resulting in a moderate overshoot level. This strategy is limited in terms of further improvements, as lowering the temperature of the first dwell or reducing its duration results in more energy release during the ramp and second dwell increasing the overshoot further, whilst increasing the temperature or duration of the first dwell results in manifestation of a higher overshoot at its end. Modification of the second dwell temperature also has a negative effect as reducing it results in manifestation of the overshoot at a lower tool temperature and increasing it in its occurrence during the ramp.

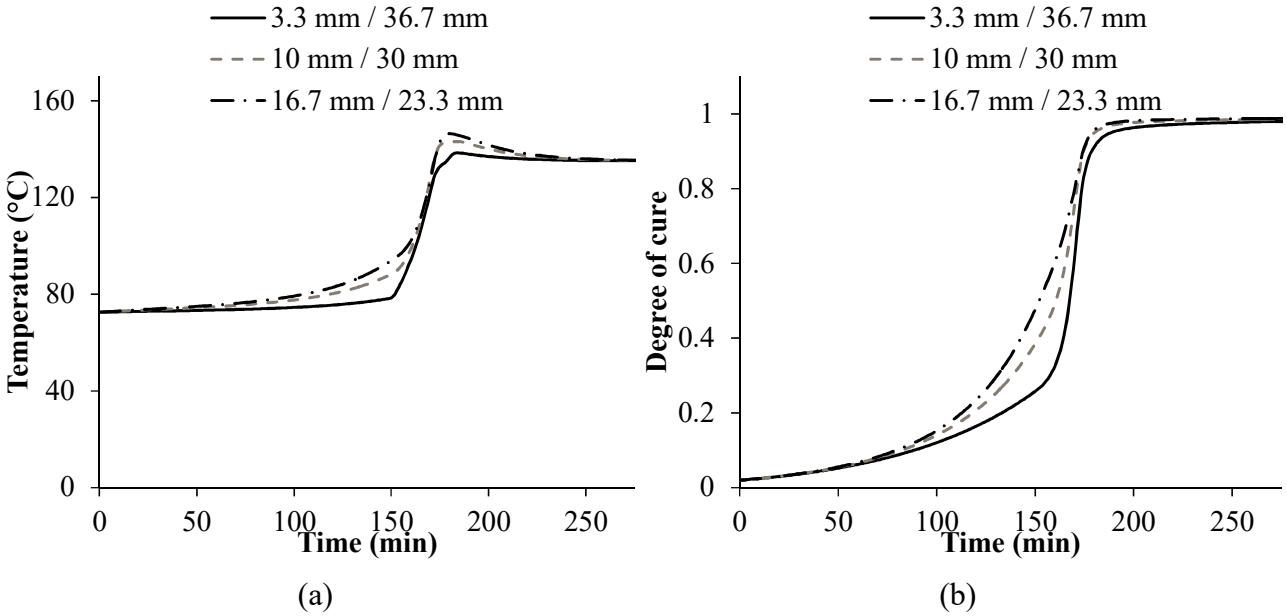
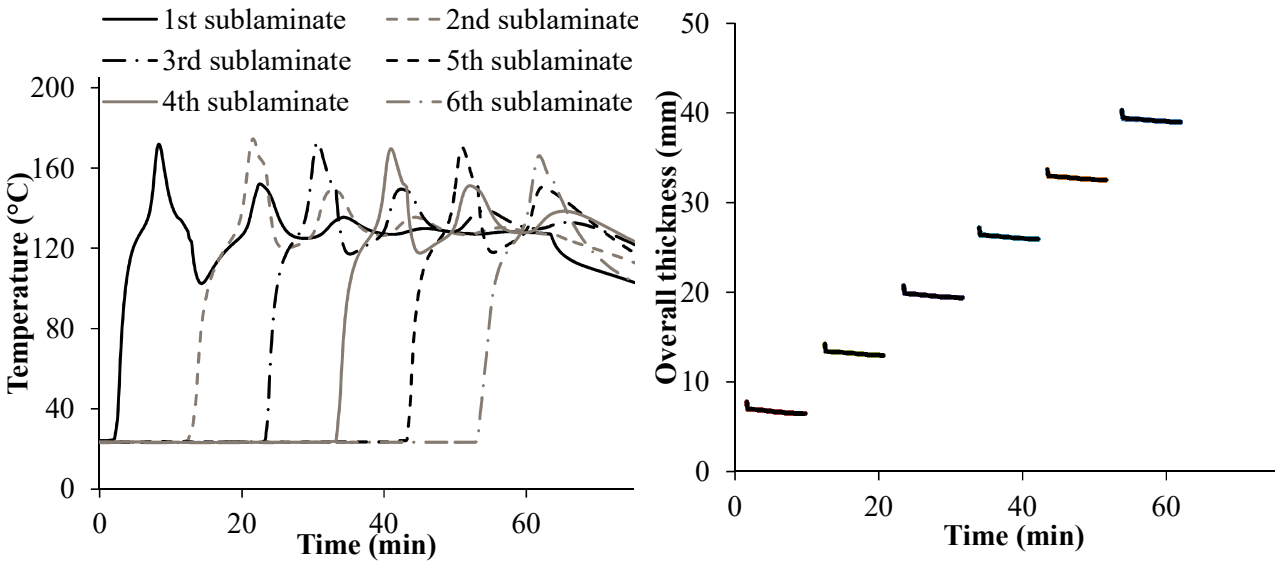


Fig. 9 Simulation of conventional cure using moderate optimised conditions (point A in Fig. 8): (a) temperature at locations corresponding to mid-thickness of sublaminates; (b) degree of cure on top layer of sublaminates

3.4.4. Process trial

The results of monitoring of temperature and compaction during the LbL process trial are illustrated in Fig. 10. The temperature evolution has the periodic characteristics predicted by the simulation (Fig. 7). The level of temperature overshoot at sublaminate mid-plane is 45 °C, which is very close to the value predicted by simulation (50 °C). The cyclical heating of each layer after its placement goes through lower minima (about 20 °C in the second cycle) and a slight lag is observed for lower sublaminates accompanied by broadening of the overshoot peak. These results show that the simulation of the cure is valid and the result obtained with respect to process efficiency as reported in Fig. 8 hold true. The completion of the cure in the process trial is slightly lower than that predicted by the simulation (67 instead of 70 min) as a result of variations of the cooling stage duration of the processing cycle during the trials. The compaction monitoring results presented in Fig. 10.b also confirm the outcomes of the simulation. Compaction occurs very quickly upon application of pressure on each sublaminate, whilst a final thickness of around 6 mm is reached in each layer corresponding to a fibre volume fraction of about 60%.



(a) (b)

Fig. 10 LbL process trial: (a) temperature at mid-thickness of sublaminates; (b) thickness evolution

The quality of the material produced (Figs. 11a and 11.b) using the LbL process was investigated by microscopy. The micrographs in Figs. 11.c-11.f compare the morphologies of the LbL material with material produced conventionally in the press. In both cases the matrix has a dual phase morphology. Domains appearing dark in optical micrographs (Figs. 11.c and 11.e) correspond to a modifier rich phase. This is shown clearly in the SEM micrographs in Figs. 11.d and 11.f, where the dark regions of optical micrographs appear as light coloured particles that seem to deform during sample preparation. These domains are elongated, possibly as a result of consolidation, whilst their aspect ratio and size are greater in conventionally processed material possibly as a result of the UD layup of this material and the moderate temperature history. Microscopy does not reveal any significant levels of porosity in the LbL material; the overall picture is very similar to that of the conventionally processed composite. These results show that the quality of the LbL material is similar to that obtained in conventional processing. The consolidation levels achieved are appropriate and voids have been removed. Small changes and potential refinement of the dual phase morphology of the epoxy matrix can be explained by the difference in thermal history and layup and are not expected to cause significant variations in resin dominated properties of the material.

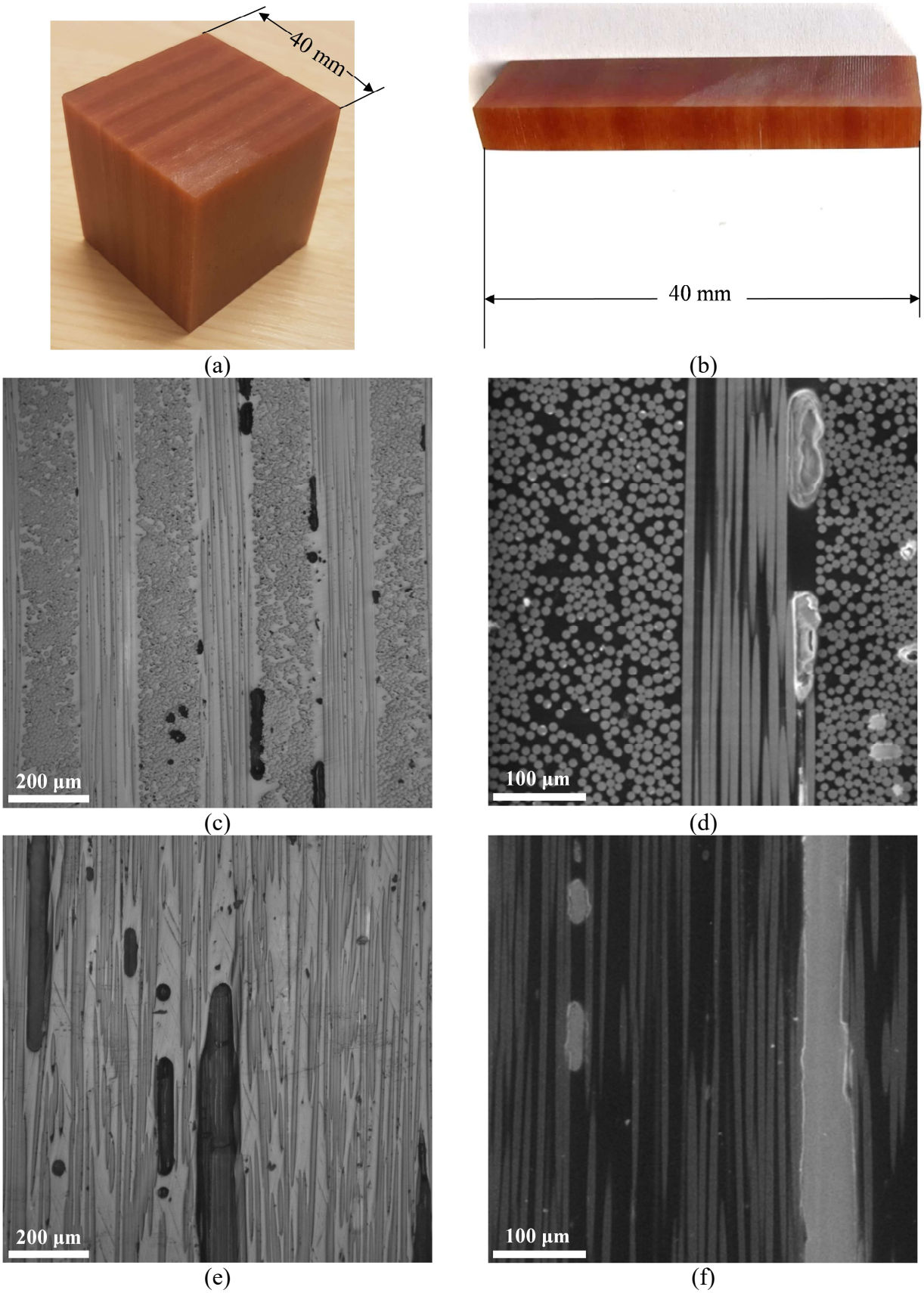


Fig. 11 Product of the LbL process and comparison with conventional curing: (a) LbL laminate 40 mm cube, (b) section across the thickness of the LbL laminate; (c) optical micrograph of LbL laminate; (d) SEM micrograph of LbL laminate; (e) optical micrograph of conventional UD laminate; (f) SEM micrograph of conventional UD laminate

3.5. Conclusion

The simulation and experimental results of this study showed that the LbL process concept put forward in this investigation is successful. The process can deliver thick and ultrathick laminates produced in an additive manner. The quality of laminates obtained with the LbL process matches that produced by conventional processing whilst the degree of pre-cure reached in a sublaminate after its placement and consolidation can be adjusted to a level around the gelation degree of cure of the matrix to achieve interfacial properties equivalent to those of standard material. The LbL process brings about significant efficiency benefits as cure process duration can be halved for equivalent temperature overshoot, whereas temperature overshoots can be reduced by 75% for equivalent process duration. The process duration benefits are intensified further by the fact that in the LbL process the consolidation stage is merged with cure, which for cases where deposition/consolidation needs a similar amount of time as cure, implies an overall process duration benefit of about 75%. Furthermore, the LbL process can operate in conditions that cannot be reached by conventional processing and can be used for the production of thicknesses that are impossible to manufacture using standard methods.

The potential of the LbL process stretches well beyond thick and ultrathick structures. Its capability for fast curing and for merging the consolidation with cure can find uses in the context of automated fibre placement (AFP) and automated tape laying (ATL). The opportunities offered by designing a tailored LbL process with sublaminate thickness, temperature and cycle duration treated as optimisation variables can increase efficiency and render feasible the processing of extra-large structures. The methodologies developed here require expansion for simulation to address three dimensional complexity and additional process physics around the build-up of process stress and for manufacturing to incorporate integration with robotic placement heads aiming at using the LbL process for complex curved geometries.

3.6. References

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4. Core project proposal plan

Advancement of the concept proven in the feasibility study will be sought through a Future Manufacturing Research Hub Core project. The overall aim of the proposed work is to establish the LbL process at the scale and level of complexity required for advanced applications to composite structures and to develop the scientific and technological tools necessary for the future implementation of the concept.

4.1. Main Objectives

The feasibility study has shown that the challenges of high speed consolidation at aggressive conditions and controlling the level of cure to achieve appropriate levels of adherence between layers/sub-laminates can be addressed in simple planar geometries. The translation of this to components involving 3D features, local placement of material, curvature, large dimensions and multi-material combinations will enable the process to deliver its high rate benefits in the industrial context. This will be achieved by addressing the following high level objectives each corresponding to a pillar of activity within the project:

- ✓ Process optimisation based on 3D coupled (consolidation/cure/thermomechanical) numerical simulation to achieve maximisation of interfacial toughness and minimisation of process duration.
- ✓ Investigation of LbL process implementation within the whole process chain, including defect generation due to ultralow viscosity, ply drop offs, gaps, curvature and their control.
- ✓ Demonstration of the process capabilities and applicability based on lab/pilot scale LbL implementations.

4.2. Approach

The development of the LbL process will be carried out within the three pillars of activity corresponding to the project objectives, addressing scientific, processing and technological challenges (Fig. 12).

Scientific developments, which will provide the tools needed to predict and control material behaviour comprise:

- ✓ Fully-coupled 3D heat transfer, consolidation, and residual stress model interfaced in an open source programming language.
- ✓ Material property characterisation for rapid processing technologies involving snap cure materials, including degree-of-cure, compaction, and rheology.
- ✓ Optimisation of adhesion and residual stresses between layers of similar/dissimilar materials.

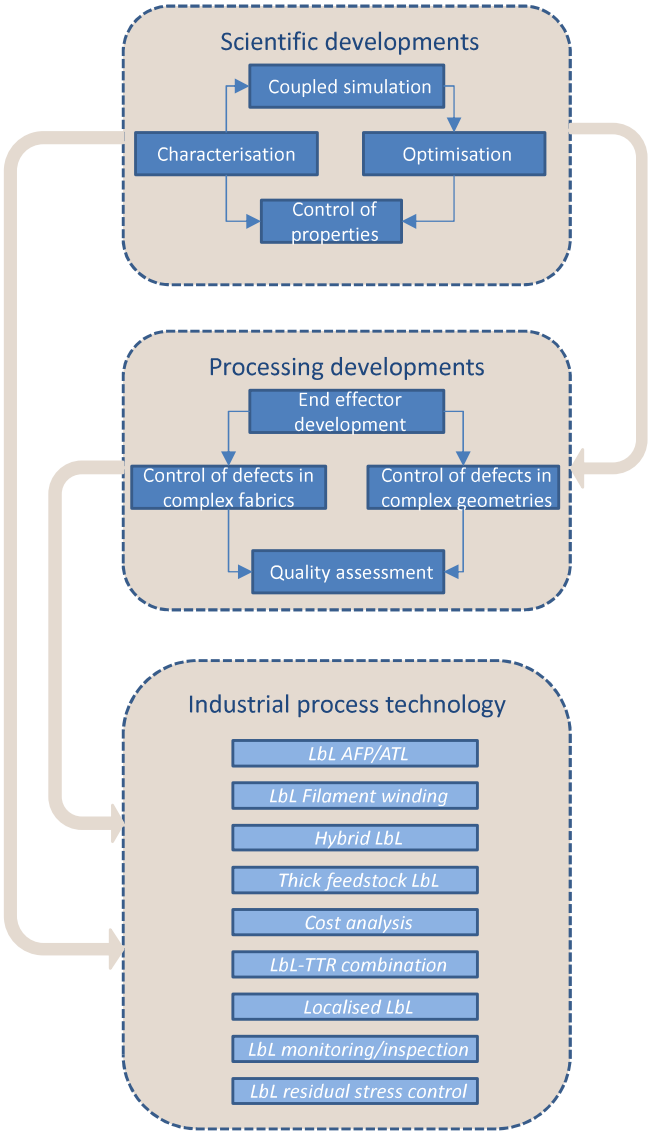

Fig.12 Project approach and interdependencies

- ✓ Control of toughness/wet T_g through management of polymer morphology and material state.

Processing developments will provide understanding the influence of material inputs and process conditions, and develop the tools allowing controlling final part quality by:

- ✓ Development of end effectors for automated processes and flexible bags for complex geometries capable of processing low viscosity materials and achieving requisite temperature control.
- ✓ Identification of process conditions appropriate for eliminating defects (voids) in areas with gaps, overlaps, and ply drops.
- ✓ Investigation of the processing of complex/curved geometry laminates and core sandwich structures and elimination/minimisation of corner thinning, ply bridging, and core crushing at close-outs.
- ✓ Evaluation of quality of manufactured parts by microstructural observations and mechanical testing.

Process technology will up-scale the scientific and processing technologies to the level required for manufacturing of advanced composite structures. Activities include:

- ✓ Integration of LbL curing in AFP/ATL processing of complex parts.
- ✓ Processing of thick/ultra-thick filament wound structures without a secondary cure stage.
- ✓ LbL processing of hybridised preforms of thermoplastic and thermoset polymers offering spatially tailored properties.
- ✓ LbL processing of multi-orientation thick feedstocks for large structures.
- ✓ Cost modelling of LbL methods and comparison to conventional processes over a wide range of component geometries and types.
- ✓ LbL processing with through-thickness-reinforcement, such as z-pins, for interface enhancement.
- ✓ Localised LbL curing to stabilise complex preforms and eliminate forming defects.
- ✓ In situ Layer by Layer monitoring and inspection.
- ✓ Control/tailoring of residual stress in LbL processing to achieve desirable effects, such as pre-stresses.

4.3. Partnership

Engagement with potential industrial partners is ongoing. The team of industrial partners envisaged will comprise end users supporting process technology developments focused on relevant application areas, equipment and software providers enabling process developments and Catapult Centres enhancing the innovation potential of the LbL concept and facilitating its future adoption. In addition, partnerships with specialist academic groups are envisaged to leverage scientific developments. Discussions with the following organisations are currently taking place:

- ➢ End Users
  - ✓ Rolls Royce
  - ✓ Airbus
  - ✓ Luxfer
  - ✓ BAE Systems
  - ✓ Leonardo
- ➢ Equipment Developers
  - ✓ Coriolis Composites
  - ✓ Heraeus
- ➢ Simulation Software Developers
  - ✓ ESI
- ➢ Catapult Centres

- ✓ National Composites Centre/High Value Manufacturing Catapult
- ✓ Offshore Renewable Energy Catapult

- Academic Groups
  - ✓ University of Nantes – Heat Transfer and Energy Laboratory
  - ✓ McGill University – Structural and Composite Materials Laboratory

4.4. Resources plan

The project duration planned is three years. Funding for two full time PDRAs, one based at Cranfield and one Bristol, as well as 10% of each of the Investigators will be sought. This will be complemented by resources sought for modification and adaptation of existing equipment to enable process trials and specialised characterisation to be carried out, consumables and dissemination activities.

PhDs/EngDs will be funded/co-funded by End Users targeting relevant developments with the Process technology pillar of activities Access to specialised equipment will be contributed by Equipment Developers and to simulation software by a Software Developer. Potential co-funding of PhDs with external academic Groups will also be considered during preparation of the proposal. Institutional support will comprise co-funding of Doctoral students as well as internal MSc projects supporting activities of the project. Catapult Centres will provide guidance with respect to innovation aspects of the work and also support with access to equipment where needed. All partners will also contribute with participation in the steering committee, attendance of technical and management meetings and access to specialists providing expert advice on specific topics around processing developments and technology.

Requested funding will be approximately £600k with the targeted overall project value reaching around £1.2 MM.