MANUFACTURING OF COMPLEX SHAPE COMPOSITE PARTS THROUGH THE COMBINATION OF PULL-BRAIDING AND BLOW MOULDING

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Keywords: Blow moulding, Braiding, Pultrusion, Two-step-curing

ABSTRACT

The goal of the funded project PulForm is the development of a reliable and economical processing technology for the production of hollow thermoset CFRP parts of complex shape. This is achieved through the combination of pull-braiding, i.e. an assembly consisting of a braiding and a pultrusion machine, and blow moulding processes.

The resin is a hybrid two-step-curing thermosetting resin. In the first reaction step (polyurethane addition reaction), taking place in the pultrusion die at 100 °C, a B-stage of rubber-like consistency is reached. In the second reaction step, the B-staged part is heated to a temperature above 100 °C, triggering a radical polymerization reaction to full curing of the matrix. Selection of an appropriate resin formulation was done by means of rheology measurements during cure and picture frame tests on braided preforms wetted out with resin and brought to a B-stage state.

In the first processing step, a tube (outer diameter 58 mm) with a fiber structure consisting of layers of unidirectional carbon fiber rovings and braided layers is continuously produced by pultrusion. The braided layers are produced in-process by a braiding machine assembled upstream of the pultrusion line. The resin is injected into a closed impregnation chamber attached to the heated pultrusion die.

In the second processing step, the pultruded B-staged tube is placed inside a heated mould mounted to a press. With the mould closed, pressurized air is blown into an inflatable bladder, pressing it against the tube and the mould inner walls, and thus giving the part its final shape. The moulding process step is evaluated through trials in a mould with a conical expansion geometry.

The processing-related material behaviour is evaluated through curing of coupon layers under pressures similar to the applied in blow moulding and subsequent mechanical testing (mode I and mode II interlaminar fracture toughness).

1 INTRODUCTION

One of the main scientific and technological challenges for the introduction of carbon fiber reinforced composite parts (CFRP) in series applications with medium and high production volumes (especially for the automotive industry) is the development of robust and highly automated manufacturing technologies. The aim of the German public founded Research Project PulForm is the development of a resource and energy efficient, economical manufacturing technology for hollow parts by chain-linking pultrusion, in-line-braiding, blow moulding and final machining to a flexible, quality assured and automated manufacturing process chain. The major processing steps are shown in the schematic representation (Figure 1).



Figure 1: Schematic representation of the PulForm process chain

Pultrusion is a simple and cost-effective processing technology for the continuous production of fiber reinforced composite profiles. By this processing method, the reinforcement material is continuously wetted out by liquid resin and pulled through a heated mould (usually called pultrusion die) where the resin gels and cures. The mould inner cavity imparts the profile final shape. Impregnation of the reinforcing fibers is traditionally accomplished by pulling them through a resin bath [1]. The ease of wetting by this method is contrasted by some inherent disadvantages, such as: limitation of matrix choice to resins of relatively long pot life at room temperature; quality assurance issues; and exposure to volatile chemical components at the working place. These issues are the main drive to development of closed injection and impregnation chamber which is either directly attached to the die, or machined directly as part of a pultrusion die designed for that purpose [2].

Reinforcement in pultrusion consists primarily of unidirectional fibers. Depending on the application, continuous filament mat (CFM) layers, as well as so called complex reinforcements (composed of a combination of chopped strand mat and/or surface veil and woven or non-crimp fabric layers) can also be processed to improve mechanical properties in directions other than the profile main axis of orientation. Another promising alternative is the coupling of a braiding machine upstream of the pultrusion line for the production of braided layers which are fed inline to the pultrusion die. Main advantages in the latter case is the cost effectiveness, as no intermediate steps for the production of textile semi-finished products are needed, and a seamless closed reinforcing layer for a hollow profile, i.e. without overlaps between two adjacent planar textiles [3].

Despite the advantages of the pultrusion process, the limitation in product shape has hindered a more spread use in branches such as the automotive, where integration of CFRP parts into geometrically complex assemblies requires freedom of design to complex 3D shapes. This limitation is overcome in the PulForm process by chaining the continuous pultrusion process for the production of B-staged preforms and a batch postforming process, i.e. blow moulding, to produce a hollow part of complex 3D geometry.

The resin is a formulated 2-component reactive system based on the Daron[®] turane chemistry (DSM Composite Resins). Through the mixing of processing additives, the curing cycle can be adjusted to fit both processing steps. The first reaction step is a urethane polyaddition reaction, consisting in chain polymerization between isocyanate molecules and hydroxyl functional groups present in the resin backbone chains. The initially liquid resin goes through a gel state to a B-stage condition, in which the material is not tacky but still flexible (rubber-like consistency). This reaction step takes place in the pultrusion die, through which the reinforcement material, after being wetted out by resin, is continuously pulled. The die is heated to temperatures between 80 °C and 100 °C.

By increasing the temperature above 100 °C a radical polymerization is initiated, leading to fully curing of the matrix. This second reaction step consists of cross-linking of the polymer chains and polystyrene. This second reaction step takes place in a postforming mould.

Blow moulding processes are very common for thermoplastic parts. Processes based on prepreg materials which are formed by pressing against an inner cavity by an inflatable bladder are used in the production of sport articles (e.g. bicycle frames). A similar process is also described for preforming a dry textile and subsequently injecting a resin system and curing, as a variation of the resin transfer moulding (RTM) process for the production of hollow bodies with high fiber volume content [4].

2 MATERIALS AND METHODS

2.1 Resin system

The resin system is a 2-component reactive system. The resin is a prepolymer named Daron ZW 015864 (DSM Composite Resins, Zwolle, NL), which is diluted in styrene. To adjust the resin flexibility in B-stage, another reactive resin additive named Daron ZW 8117 (also from DSM) can be added. A catalyst is added to increase the reaction rate in the pultrusion die, as well as an internal mould release to reduce the adhesion of resin on the die wall. An inhibitor is also added to minimize radical release and prevent an early onset of the second reaction step in the pultrusion die. The hardener for the B-staging reaction is a di-isocyanate (MDI). Organic peroxide is added to the hardener, which promotes radical reaction initiation at temperatures above 100 °C.

2.2 Rheology measurements

For selection of an appropriate resin formulation and adjustment of mixing ratios of processing additives, rheology measurements were performed on different formulations during cure in a rheometer (Anton Paar Physica MCR 501) with disc-plate configuration in oscillation modus. The frequency was set to 1 Hz and the strain was adjusted according to the reaction progress. At 80 °C a strain of 1 % was set (deflection angle 0,8 mrad), and at 160 °C, the strain was 0,1 % (deflection angle 0,08 mrad). As measurement results, a dynamic modulus of shear is obtained, i.e. a storage modulus G' and a loss modulus G'', from which a modulus of the complex viscosity can be calculated [5]. According to the Cox-Merz rule, the modulus of the complex viscosity agrees relatively well with the apparent viscosity of viscoelastic materials at the same shear rate [6].

The setup for temperature evolution was chosen such as to emulate processing conditions. The material is subjected during a rheology measurement to the temperature cycle shown in Figure 2(a). The start and end temperature is 20 °C, all heating and cooling down steps are applied at a rate of 5 K/min.

2.3 Picture frame tests

To evaluate the processability of the B-stage preform in the postforming process step, a series of picture frame tests were run on a universal testing machine (Hegewald & Peschke 250 kN). The sample geometry was 200 x 200 mm². Measurements were performed on a single layer of biaxial braided reinforcement structure (made of carbon fiber rovings of the type Toho Tenax STS40 F13 24k), as well as on layers of the same braided structure wetted out with two different resin formulations and brought to B-stage. Wetting out was achieved by vacuum infusion and B-staging occurred at room temperature over 12 hours. Picture frame testing was performed for the wetted out samples at room temperature and at 80 °C. A curve shearing force vs. shearing angle is obtained by the method.

2.4 Interlaminar bonding testing

In order to evaluate the quality of interlaminar bonding after postforming, a series of testing of mode I (G_{Ic}) (according to ASTM D 5528-01)) and mode II (G_{IIc}) (according to DIN EN 6034)

interlaminar fracture toughness was performed. For that purpose, stacks composed of various layers of a unidirectional (0°) non crimp fabric (300 g/m², made of carbon fiber rovings of the type Toho Tenax STS40 F13 24k, Haufler composites, Blaubeuren, Germany) were wetted out by vacuum infusion, and left to B-stage over 12 hours. The preforms were then pressed and cured under defined pressure levels. Heating platens of the hydraulic press were set to a temperature of 130 °C. The applied pressure cycle was 15 min. The pressure set point was reached in all cases in less than 1 minute. The plate configurations are shown in Table 1.

Plate Identification	Forming Pressure [bar]	Sample Thickness after pressing [mm]	Description Preform composed of 10 NCF layers wetted out in one vacuum bag (polymer film insert placed before infusion)			
40_d	40	2,40 ± 0,03				
4_m	4	$2,70 \pm 0,08$	Preforms composed of 5 NCF layers wetted			
10_m	10	$2,66 \pm 0,02$	out in one vacuum bag, staking of 2 preforms with polymer film insert placed between			
40_m	40	$2,45 \pm 0,02$	them immediately before pressing			

Table 1: Plate configurations for interlaminar bonding testing specimens

After pressing, the plates were cut and the samples bonded to loading blocks to a double cantilever beam (DCB) specimen with a length of 250 mm, allowing for G_{Ic} testing, and subsequent cutting of an end notched flexure (ENF) specimen for G_{IIc} testing. Data analysis of G_{Ic} was performed according to the three methods described in ASTM D 5528-01: the modified beam theory (MBT), the compliance calibration method (CC) and the modified compliance calibration method (MCC).

3 EXPERIMENTAL

3.1 Resin analysis

In order to find an appropriate resin formulation combining the requirements for the first B-staging reaction step (fast enough B-staging to allow continuous pulling through the pultrusion die, matrix with low or no tackiness, enough rigidity to withstand the pulling mechanism), and the second curing reaction (enough flexibility to allow for blow moulding), a series of rheology measurements was performed on different formulations according to the procedure described in section 2.2. Specifically the ratio of ZW 015864 to ZW 8117 was varied, resulting in the curves for the modulus of viscosity (Figure 2(a), previously published in [8]).



Figure 2: (a) Comparison of viscosity curves for different resin formulations; (b) Storage and loss moduli curves during the first section of the measurement (up to 80°C isothermal plateau).

In the beginning of the measurement, the viscous character of the resin prevails, i.e. the loss modulus is higher; as the reaction starts and proceeds, the storage modulus, corresponding to the elastic portion of the matrix increases, until it crosses and becomes higher than the loss modulus. The crossing point between the moduli is often used as an estimation of gel point [7] and is shown for the formulations No. 1 and No. 2 in Figure 2(b).

Due to the lower reaction rates, the formulations No. 3 and No. 4 were not considered for processing trials. This is clearly seen by the viscosity curves, where after 30 min at a constant temperature of 80 $^{\circ}$ C, the samples had not yet reached a plateau, i.e. the B-staging reaction was not completed.

As an additional analysis tool for formulation selection, a series of picture frame tests were performed as described in section 2.3. A comparison was made between a single layer of dry braided material, a single layer of braid wetted out with formulation No. 1 and single and double layers wetted out with formulation No. 2. The force needed to shear a wetted out braid was much larger than for the dry braid. Furthermore, the layer with formulation No. 1 did not show reproducibility for a pure shearing behaviour; the specimens were very stiff and showed buckling in many tests immediately after the onset of force application. Therefore, no reproducible curves could be recorded for formulation No. 1. Formulation No. 2 showed on the other hand a reproducible behaviour and could be submitted to pure shearing. The curves are shown in Figure 3 (previously published in [8]). Reaching of the lock angle is represented in the graphs by the curve peak, after which the sample starts to buckle, thus reducing the applied force necessary to further shearing. A clear reduction of needed shearing force is observed by comparing the curves for a single layer shearing at room temperature (red dotted curve) and at 80 °C (blue curve). It is therefore advantageous to postform the material at an increased temperature between 80 °C and 100 °C before the curing reaction is triggered.



Figure 3: Curves of shearing force vs. shearing angle for picture frame tests.

From the analysis presented above, the formulation No. 2 was chosen as the most appropriate for processing trials, as it presents more flexibility as the resin No. 1, but at the same time reaches a B-stage in a relatively fast reaction time.

3.2 Pull-braiding process

A pultrusion machine (Px500-10T, Pultrex Ltd., Essex, UK) was used for pulling the reinforcement material through the pultrusion die. The die has a cavity such as to produce tubes with an outer diameter of 58 mm. The inner diameter can be set to 2,5 mm or 3,0 mm by means or different mandrels. The die was manufactured from tooling steel according to the state of the art in pultrusion processing. The die has 1 meter in length and is heated by means of clamping heating plates to the outer surfaces of the die. Three heating zones are set to a temperature of 100 °C. The temperature

along the die is measured in different points through thermocouples inserted into holes with ends close to the die cavity.

The pultrusion die was adapted to closed injection and impregnation. For that purpose, two different injection chambers were machined and mechanically fitted to the entrance of the die. One of the injection chambers has a "tear drop" geometry; the other has a conical inlet. Schematic drawings of the chambers are shown in Figure 4. The mandrel was also adapted to allow resin injection from the centre to the inner layers of material, and was mounted to a mandrel holder upstream of the braiding machine.



Figure 4: Schematic drawing of the injection and impregnation chambers attached to the pultrusion die. (a) Tear drop geometry; (b) Conical geometry

A metering and mixing unit (Nodopur VS, Tartler GmbH, Michelstadt, Germany) is used to mix the 2 resin components to a defined mixing ratio and inject the formulated resin into the chamber. The unit is based on precision gear pumps feedback controlled by volume meters. A constant mass throughput was used in all trials.

The braiding machine (RUh 2/60/84-120, August Herzog GmbH & Co. KG, Oldenburg, Germany) is a double ring unit, i.e. capable of producing two layers of braided material at the same time. The inner braiding ring supports up to 60 spools and the outer ring up to 84 spools. By setting the rotation speed of the inner and outer braiding rings, the braiding angles can be adjusted according to the linear pulling speed. A braiding angle of 30° was set for all trials. Additionally to the braiding layers, rovings can be fed to the braiding unit such as to form up to 3 unidirectional layers (0°) as inner (24 positions), middle, i.e. between the braid layers (36 positions) and outer layer (48 positions). The machine was positioned upstream of the pultrusion die, the centre of the braiding ring was aligned to the centerline of the pultrusion die.

Carbon fiber rovings of the type Sigrafil C30 50k (SGL Group, Meitingen, Germany) were used to build unidirectional 0° layers, and STS40 F13 24k (Toho Tenax Europe GmbH) were used to build the braid layers. The laminate layups investigated in the trial series are described in Table 2.

		Layup 1	Layup 2	Layup 3	
	Outer UD (0°)	34 rovings	-	-	
Layers	Outer Braid (±30°)	42 spools	84 spools	84 spools	
	Middle UD (0°)	36 rovings	44 rovings	72 rovings	
	Inner braid (±30°)	30 spools 30 spools		30 spools	
	Inner UD (0°)	24 rovings	24 rovings	24 rovings	
Laminate thickness		2,5 mm	2,5 mm	3,0 mm	
Fiber volume content (target value)		58,5 %	58,0 %	57,6 %	
Fraction of UD		70,0 %	61,0 %	52,3 %	

Table 2: Laminate layups applied in pull-braiding trials.

The pulling speed set point was 0,5 m/min in all trials. Due to the flexible nature of the B-staged preform, the material has to be supported by a mandrel in the region where the profile is clamped. This leads to pauses during processing, such that the pulling speed cannot be maintained constant throughout the trial.

One of the major challenges experienced in the pull-braiding trials was achieving a good impregnation quality across all layers. Especially the UD-layers are more difficult to fully wet out due to the high degree of anisotropy in the permeabilities along and crosswise to the fiber orientation. To achieve this, the injection chambers are supposed to be full with resin at all times, regardless of whether the material is being pulled though the die or not. For the tear drop design, this means finding an adequate cross sectional area in the chamber inlet region such that enough pressure is built up in the chamber when material is being pulled, and on the other hand, resin can flow out of the chamber inlet when the process is paused without pressure build-up in the chamber (see Figure 4(a)). The trial configurations are listed in Table 3, and pictures of trial assemblies are shown in Figure 5.

	Trial Setup								
	Ι	II	III	IV	V	VI	VII	VIII	
Laminate layup	1	1	1	1	2	3	3	3	
Resin flow rate (g/min)	150	150	150	150	150	170	170	170	
Injection chamber geometry	Tear drop	Conical							
Cross section ratio chamber inlet / die	1,00	1,07	1,09	1,40	1,09	1,18	1,18	-	
Positioning braiding machine (relatively to chamber inlet)	Far	Far	Far	Far	Close	Far	Close	Close	

Table 3: Different setups investigated in pull-braiding trials.



Figure 5: (a) Trial assembly with tear drop injection chamber and braiding machine far from chamber inlet (setups I through IV); (b) Trial assembly with conical injection chamber and braiding machine close to chamber inlet (setup VIII).

Figure 6 shows typical curves of resin injection pressure (measured for one resin component at the gear pump discharge) against time during continuous pulling procedure for different trial setups listed in Table 3. Setups II and III are not suitable for stable processing, as a constant resin throughput leads to pressure build up inside the chamber. A comparison of the curves from setups VI and VII shows that the different positioning of the braiding machine relatively to the injection chamber is significant to processing; while stable processing was achieved in both setups, the pressure level is higher if the braiding machine is close to the chamber. All other parameters for setups VI and VII were identical.



Figure 6: Curves of resin injection pressure for different trial setups (see Table 3).

Positioning of the braiding machine close to the injection chamber also proved to facilitate impregnation in the trials with the tear drop chamber (setups V and VII). Since the outer braided layer is forced against the chamber wall in the entrance region, resin excess is forced to flow though the layers before flowing out through the chamber inlet. In these trial setups, full wetting out could be observed reproducibly throughout the trials.

For the conical chamber, successful processing is dependent on the positioning of the reinforcement materials at the inlet. This geometry has been shown to work well with UD-only reinforced profiles, where a cover plate with holes can be bolted to the chamber, such as to distribute the rovings uniformly throughout the entrance cross section. For complex laminate layups including braided layers, this cannot be done. In order to force the individual layers to enter the die spread out from each other, the braiding machine was positioned at such a distance to the injection chamber as to allow the outer braided layer to close itself when already inside the chamber. The closing position of the inner braided layer is defined by a guide ring (see Figure 4(b)). However, this measure did not lead to stable processing and sufficient wetting out.

3.3 Blow moulding process

In the second processing step, the pultruded B-staged tube is placed inside a heated mould mounted to a press. A silicone bladder is fitted through the tube. With the mould closed, pressurized air is blown into the bladder, pressing it against the tube and the mould inner walls, and thus giving the part its final shape. The mould geometry has a conical expansion (angle 5°) starting from a cylindrical region of diameter 60 mm. The trial assembly is shown in Figure 7. The mould is fitted to a hydraulic press or mould carrier. A heating unit (heating medium pressure water) is coupled to the mould.



Figure 7: Postforming mould with cylindrical expansion.

Processing parameters (temperature and pressure inside the bladder) have to be chosen such as to allow the B-staged preform to deform before the radical polymerization starts. For that purpose, a variothermic process cycle was set according to Figure 8(a). Taking into consideration the results of picture frame testing (section 3.1), tempering of the preform to a temperature of 80 °C – 90 °C in the closed mould is set for a time interval of 5 min, then pressure is applied to a constant pressure increase ramp, while the heating temperature is increased simultaneously.

For recognition of material contact to the mould inner walls, a contact sensor based on a chip antenna was developed. The sensor eigenfrequency is changed when contact to the B-staged material takes place. The sensor signal is processed to an analog voltage output. A sensor was fitted in the mould in the conical region at a distance of 60 mm from the transition between cylindrical and conical regions in the mould. This corresponds to a local diameter of 70 mm. A typical sensor signal is shown in Figure 8(b), for the time span marked in the red dotted square in Figure 8(a). In this case, the preform touches the sensor 45 s after starting of the blow moulding step, at a pressure of 3 bar.



Figure 8: (a) Variothermic blow moulding cycle; (b) Contact sensor signal showing moment of preform touching the mould cavity (previously published in [8]).

For preliminary blow moulding trials, preforms with different layer configurations were produced by vacuum infusion, left to B-stage at room temperature over 12 hours and subsequently wrapped in a polymer film and stored in a freezer at -18°C, in order to hinder further reaction and diffusion of volatile components. The preforms were taken out of the freezer shortly before processing. Postformed parts are shown in Figure 9.



Figure 9: blow moulded parts from different preform configurations (a) Single layer braid with original braiding angle ±45°; (b) Single layer braid with original braiding angle ±30°; (c) Outer braided layer with original braiding angle ±45° and inner unidirectional (0°) layer.

From the analysis of angle distribution one can conclude that a small braiding angle of e.g. 30° in the preform is more adequate if the part is designed with considerable perimeter expansion in the postforming step. For a braiding angle of 45° , the part reaches a maximum local angle of around 65° (in Figure 9(a), at a diameter of approx. 70 mm) and starts to buckle. Another important finding is that, during postforming, the individual layers can slide relatively to each other. This is clearly shown in Figure 9(c). UD and braid layers were cut to the same length before blow moulding; through shearing of the braid layer, the length of this layer is reduced and the UD layer stands out. The same sliding effect is expected to occur with preforms produced both by vacuum infusion and pultrusion, as the individual layers can be peeled off of each other. While this is an advantageous feature for the postforming process, it raises the question about the quality of interlaminar bonding quality after postforming. For this reason, interlaminar bonding testing was performed (section 2.4 and 4).

4 DISCUSSION AND OUTLOOK

Mode I and II interlaminar fracture toughness testing was performed on the samples described in section 2.4. The formulation No. 2 was used for wetting out of preforms (see Table 1) by vacuum infusion. Results are shown in Figure 10.



Figure 10: Results for Mode I and II interlaminar fracture toughness.

From the results, a clear effect of pressure applied to the B-staged preforms during curing on the interlaminar bonding quality, as evaluated from G_{Ic} and G_{Ilc} , can be verified. Good interlaminar

bonding is also achieved even for the relatively low pressure of 4 bar. Results reported in the literature for epoxy-carbon fiber composites range between 100 and 400 J/m² [9]. The standard deviation in G_{Ic} results is also reduced for the higher pressure sample, indicating more uniform bonding between layers. For further process development, a moulding pressure of 40 bar is therefore recommended.

Sample 40_d showed unexpectedly the lowest values for all tests. One reason for this might be the difficulty of completely wetting out the fabrics with resin in the region near the polymer film, if the film is already placed between fabric layers before infiltration. In this case, conventional vacuum bag infusion could not be applied, a VAP (vacuum assisted process) method with several injection gates was employed to wet out the preform instead.

Further process development is currently being carried out for the blow moulding step. One goal is to investigate the postforming geometric constraints of a preform by means of different geometric features. A mould with an S-shape was manufactured, and another one with different geometrical features (S-shape, transition from round to square, conical expansion) is currently being manufactured. Figure 11(a) shows the S-shape mould. Automated placing of the preform into this mould could be demonstrated. Due to the fast placing of the preform (in comparison to the conical mould) and beginning of pressure application, an isothermal process cycle could also be achieved. A pressure of 40 bar could be applied over various moulding cycles without damaging the bladder.

Figure 11(b) shows the geometry of the newly designed demonstration part. In the corresponding postforming mould, a total of five sensors (see section 3.3) are being mounted to recognize contact of the preform to the mould walls in different positions. Additionally, two dielectric sensors (Netzsch-Gerätebau GmbH, Selb, Germany) will be mounted to detect reaction evolution and assist in process cycle optimization. The basis for this on-line monitoring technique is the characterization work carried out by [Chaloupka et al., proceedings of the ICCM20, Copenhagen, July 19-24th 2015].



Figure 11: (a) S-shaped mould with postformed part and bladder (Voith Composites); (b) Geometry of demonstration part showing different geometrical features.

9 CONCLUSIONS

In this work, the process development of pull-braiding and blow moulding for the manufacturing of complex hollow parts based on a two-step curing resin system is described. A process-related characterization and selection of an appropriate resin system by means of rheology and picture frame test was carried out. Development of the pull-braiding process targeted on obtaining sufficiently wetted out B-staged preforms applying a closed injection and impregnation technique. One of the trial assemblies was shown to deliver reproducible results in terms of preform quality and processing stability. Process development of the blow moulding step focused on the definition of a process cycle, in terms of temperature and pressure, to facilitate postforming. Further development in this process step will focus in automation and in-line process monitoring to optimize processing cycles. Due to the observed behaviour of individual layers of material peeling off of each other while in B-stage condition, a testing series was performed to investigate the quality of interlaminar bonding after postforming under different pressures. The results showed a strong correlation of interlaminar fracture

toughness and the applied pressure, and that good quality composites can be obtained for the forming pressure levels investigated.

ACKNOWLEDGEMENTS

This research and development project was funded by the German Federal Ministry of Education and Research (BMBF) within the Framework Concept "Research for Tomorrow's Production" (funding number 02PJ2035) and managed by the Project Management Agency Karlsruhe (PTKA). General start-up of the project group, facilities and key technology equipment in Augsburg is funded by the Region of Bavaria; City of Augsburg; BMBF and the European Union (in the context of the program "Investing In Your Future" – European Regional Development Fund). The author also thanks the companies and other research institutes involved in the project: Audi AG, Arotec GmbH, DSM AG, fem-solutions, Mapal KG, Maus GmbH, Technokon GmbH, Voith Composites GmbH & Co. KG, DLR-ZLP, and the Institute of Aircraft Design of the University of Stuttgart. The author is responsible for the contents of this publication.

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