Flow-speed-controlled quality optimisation for one-shot-hybrid RTM parts

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ABSTRACT
This work describes a model-based methodology to improve the bonding quality between the metal and composite constituents of one-shot-hybrid resin transfer moulding (OSH-RTM) parts. In order to reduce void induced defects in the interface an ideal flow front velocity needs to be achieved. This ideal flow front velocity is characterised by capillary rise experiments at the used carbon fibre textile. The flow front velocity during mould filling is controlled by the use of pressure sensors and Darcy’s law. Therefore, viscosity characterisation of the resin system and permeability measurements of the preform were carried out. The interface of the produced OSH-RTM roof bar for a car is tested on a component test rig imitating the load of a side impact at a car. A t-test was carried out to prove that the flow-speed-controlled injection strategy is advantageous compared to a constant mass flow injection by means of a higher maximum load transferable by the interface of the hybrid part.

KEYWORDS
Resin transfer moulding; hybrid materials; model-based processing; void reduction; interface strength

1. Introduction
The desire to integrate composite materials into metal intensive industrial branches such as automotive throws up a wide range of challenges [1–3]. While the lightweight potential of composite materials is undisputed [4] they still show drawbacks in stability after impact and crash [5]. To overcome this issue a multi-material design of the car chassis can be used. However, this leads to the need of connection points between the different materials. Different methods have been developed to connect metal components and composite structures [6–10]. Nevertheless, most of the connections are still made either by rivets [11] or gluing the two components [12]. As these options necessarily add a further step in the process chain, they are inefficient and costly.
Recent research activities have shown that the gluing step can be integrated into the impregnation stage of the resin transfer moulding (RTM) process [13–15]. There, the metal component is placed inside the mould at the same time as the textile preform. Thereafter the mould is closed and the resin system is injected. The interface between the two constituents (composite and metal) is built while resin curing. Consequently, the process is called one-shot-hybrid (OSH)-RTM. The resin system works as an adhesive towards the metal component. Comparable to a classic gluing step, the surface constitution of the metal component has a big influence on the final interface strength. The maximum transferable load can be improved by mechanical structuring [16–18], chemical etching [19] or electrical discharge treatment [20] of the metal surface. At least one of these methods should be applied to reach a sufficient interface quality towards the metal constituent. The results of these works look promising, but also raise new challenges. The fact that hybrid material combinations can reach superficial material properties by combining the advantages of the single constituents [21] comes with a drawback. Especially the difference of the thermal expansion coefficient is crucial. It leads to thermal induced stresses or even deformations if the temperature is changing [22]. In the case of OSH-RTM, this means that the difference between curing temperature and the ambient temperature, where the final part is used, is resulting in residual stresses. If the interface strength between composite and metal is too low, these stresses do not only lead to a reduction of the maximum transferable load but also to failure of the bonding. Another important fact to reach a strong interface is to reduce the amount of voids in the interface zone [18]. Any void is acting as defect and therefore creates stress concentrations [23], which can lead to failure of the bonding.

At the RTM process void formation is mainly linked to the flow front velocity during resin injection [24]. Depending on the flow front velocity either micro or macro voids are built. While a too slow injection leads to macro voids, situated in between the fibre bundles, a too fast injection leads to micro voids inside the fibre bundles of the textile [25–27]. While the micro impregnation of the fibre bundles is driven by capillary forces, the macro impregnation is driven by the injection pressure. For a minimum amount of voids the two velocities should be equal [28–30]. The capillary flow-velocity can be evaluated by capillary rise experiments [28, 31–35]. As the surface polarity as well as the surface energy strongly affect the capillary pressure, such experiments need to be carried out related to the final RTM process. This means with the same textile, resin system and at the same temperature. Former studies have shown that controlling the resin flow front velocity can reduce the amount of voids in liquid composite moulded (LCM) parts [24, 35–37]. As long as the resin system is liquid and further resin is injected the produced voids are transported along the flow path [27, 35, 38]. As the injection near area is flushed by more resin the void content usually increases along the flow path [35, 39, 40]. Furthermore it is known, that a lower void content leads to higher mechanical properties [41, 42]. The voids can migrate to the surface of the composite structure as long as the resin system is liquid, then the voids can act as defect in the interface between the composite and metal of the hybrid part. Studies of adhesive bonded joints have shown that such defects are strongly affecting the bonding strength [43–46].

Darcy’s law [47] describes the flow in porous media and connects the flow velocity with the viscosity of the fluid, the permeability of the porous media and the pressure gradient. As in rigid closed moulds the optical observation of the flow front and its velocity is not possible, other methods need to be evaluated. Di Fratta et al. presented a method where pressure sensors, Darcy’s law and the continuity equation are used to calculate the shape of the flow front [48]. For simple parts using moulds with
constant cross section, line injection and constant preform properties, the application of the one-dimensional form of Darcy’s law is sufficient.

The aim of this work is to increase the interface strength of hybrid components manufactured with the OSH-RTM process. Therefore, the amount of voids located in the interface zone, which do not only reduce the bonded area but also lead to stress concentrations, should be reduced. One of the most promising ways to do so is to reach an ideal flow front velocity during resin injection.

2. Experimental work

To investigate the influence of the flow-speed-control (FSC) on the interface of OSH-RTM manufactured parts a roof bar of a car was chosen as part geometry. Fig. 1 shows the development of the part along the processing chain. It starts with an epoxy foam core, which is used to build a hybrid preform consisting of the core, a braided carbon fibre (CF) sleeve, a thin layer of glass fibres (GF) and two omega shaped metal parts covering the edges of the bar.

2.1. Implementation of the flow speed control

The FSC is based on the continuity equation and the one-dimensional form of Darcy’s law, which is shown in Equation (1):

\[ v_d = -\frac{K_x dp}{\mu dx}. \]  

(1)

The Darcy velocity \( v_d \) is the volume averaged flow velocity of the resin, which depends of the

\[ V_f = \frac{v_d}{\phi}. \]  

(2)

By knowing the parameters viscosity, permeability, porosity and ideal flow front velocity from preliminary tests, two pressure sensors with a defined distance along the flow path can be used to measure the pressure gradient during resin injection. This measurement can be used to control the resin mass flow provided by the injection unit. For this purpose, a FSC system was built [49]. Its working principle is shown in Fig. 2.

The FSC was realized on a real-time-capable programmable logic controller (PLC) to ensure a short reaction time. The mould mounted pressure sensors, and the Tartler (Germany) Nodopur VS-2K injection unit are connected via input/output (I/O) modules to the PLC. The Darcy velocity is calculated by the provided data and then compared by the PLC with the flow speed set point. Thereafter, the new set point for the mass flow is calculated and sent to the 2 K injection unit.

2.2. Prenominal tests

FSC using Darcy’s law is only possible if a sufficient set of data is available. Therefore, preliminary measurements need to be carried out.

2.2.1. Capillary rise experiments

In order to have a set point for the FSC the ideal flow front velocity must be known. Therefore, capillary rise experiments were carried out at a special test rig [31]. As the capillary flow velocity is strongly depending on the used fabric, fibre volume fraction, resin system and temperature, the experiments were carried out at the same conditions as
the OSH-RTM process. A Siltex (Germany) 3888 CF braided sleeve was cut on the side to open the sleeve and then stretched to fit the circumference of the core later used for the production of the roof bar. This ensures the right fibre orientation angle, which strongly affects the vertical capillary flow velocity. In the next step the CF sleeve was cut into pieces with dimensions of 50 x 60 mm by a Zünd (Switzerland) automated cutter. Preforms existing of three layer CF fabric were built and placed in the sample holder containing a 2 mm cavity frame to define the distance of its glass blocks. This results in a fibre volume fraction of 51%. The sample holder including the preform and the two components of the epoxy resin system (Epinal IR 77.55-A1 and Epinal IH 77.55-B1, provided by bto-epoxy (Austria)) were placed in a convection oven until they reached a temperature of 65 °C. Thereafter, the sample holder was placed in the test rig cabin and the resin system was mixed at a mixing ratio of 100:32 by weight. The measurement was started immediately to prevent the resin system to change its viscosity too much due to curing. Therefore, the sample holder was placed in the measurement cabin and the resin-system was placed beyond in a petri dish. The petri dish was moved upside by an electric lifting platform until it touched the surface of the sample. The flow front development was captured by a two-megapixel grayscale camera with fixed aperture and lens at 20 fps. After the measurement the captured pictures were used to evaluate the flow front by applying a differential image method. Fig. 3 shows a sample of the preform with the evaluated flow front position over time.

One can see that the flow front position is building up peaks and valleys along the sample width. These are linked with the structure of the braided preform. Due to local differences in the fibre volume fraction, the flow velocity is different. According to the Washburn equation, areas with low fibre volume fraction (bigger capillary radius) show a higher capillary flow velocity [50]. As voids in between the fibre bundles are built, if the capillary flow velocity is higher than the pressure driven flow during the resin injection in the RTM process, the mean of the capillary velocity peaks was taken as set point for the FSC. Fig. 4 shows an example for the evaluation method for the ideal flow front velocity. In all three measurements, seven measurement points were evaluated.

The result of the capillary rise experiments proposed an ideal flow front velocity of 5.56 ± 0.72 mm/s. According to Equation (2) this results in a Darcy velocity of 2.72 ± 0.35 mm/s.

2.2.2. Viscosity measurements
A fundamental part of Darcy law is the dynamic viscosity of the fluid. As reactive resin systems change their viscosity with respect to time and temperature, it is important to have a set of measurements, which enables one to evaluate the viscosity at any point. Viscosity measurements were carried out at different temperatures on an Anton Paar (Austria) rheometer. The evaluated time viscosity curves were used to interpolate a 3D surface to find a viscosity value for any time/temperature combination. Fig. 5 shows the extracted viscosity curve at 65 °C. In addition Fig. 4 shows the 3D surface representing interpolated viscosity data with respect to time and temperature. One can see that the viscosity is varying only in the range of 155-182 mPa s in the first 2.5 min at that temperature. This is important for the capillary rise experiments as well as for the impregnation stage of the OSH-RTM process, which takes place in this time-range.
2.2.3. Permeability measurements

The variable, which links the Darcy velocity with the pressure gradient and viscosity, is the permeability of the preform. For this set-up, the permeability tensor can be reduced to a single value representing the permeability along the flow direction. The permeability of the preform was characterized by the use of 2D in-plane permeability experiments following the radial flow principle [51–53]. The preform was built as described in the capillary rise experiments section excluding the automated cutting step. A hole with 6 mm radius was stamped in the centre point of the preform to ensure uniform distribution of the injected fluid in the vertical direction. Red coloured sunflower seed oil was used for the injection to ensure a better contrast for the camera. Analog to the composite thickness of the final component a 2 mm cavity frame was used to reach the desired fibre volume fraction. The measurement shown in Fig. 6 describes the developing flow front as well as the orientation of the permeabilities along the major axis. \( K_x \) shows the permeability along the length of the CF braid which correlates with the flow direction in the mould of the final component. The six measurements showed an average rotation angle of \( K_1 \) of 2.2 ± 2.1° with respect to \( K_x \). The permeability along the x-axis was found as 5.45 ± 0.77 E-11 m².

To evaluate if the measured value is applicable for the FSC, the components manufactured with constant mass flow were also used for permeability evaluation. As the roof bar is symmetric and two pressure sensors are available Darcy’s law can be used for the permeability calculation. For this case, the constant mass flow was used to calculate an average constant flow velocity inside the cavity by the assumption of mass and volume continuity. The viscosity of the resin system was calculated by the use of the mean temperature measured at the pressure sensor points as well as the mean arrival time of the flow front at the two sensors. The three measurements resulted in a higher permeability of 1.73 ± 0.14 E-10 m², which is higher by a factor of about 3.2 compared to the permeability value found by radial flow experiments. As this measurement is more representative for this case, this value was used for the FSC.

2.3. Manufacturing of the hybrid component

As hybrid component, a car roof bar was chosen. The preform consists of four different materials. An epoxy foam (raku-tool WB-0700 RAMPF, Germany) builds the core material. It was encased by three layer of a CF braid (3888 Siltex, Germany). To prevent contact corrosion one layer of a GF fleece (microlith ST 3022 JMC, USA) was put on top of the sand blasted omega shaped steel (1.0548 HC340LA TK, Germany) parts. Fig. 7 shows the lower mould half with the split preform above. The preform was placed in the mould preheated to 65°C.

To compare the FSC with a classic manufacturing technique two different procedures were carried out. At first three components were manufactured with a constant mass flow of 0.15 kg/min during injection. Thereafter, three components were manufactured by the use of FSC and a target Darcy velocity of 2.72 mm/s. As the Darcy velocity can only be calculated after the flow front has reached both pressure sensors, the resin injection is mass-flow controlled at 0.15 kg/min until this point. For all parts, the resin system was preheated by the injection unit to 65°C. To keep the manufactured components comparable, the injection was stopped after a total amount of 0.75 kg resin system injected. After injection, the mould was heated up to 100°C with a heating rate of 1°C/min. After a curing time of
60 min, the component was demoulded and cooled down by free convection to room temperature.

2.4. Testing of the component

To evaluate the interface quality of the hybrid component a special test rig, designed to simulate the force acting in case of a side impact, was used. Therefore, the roof bar was cut into three pieces. The two outer parts, containing the hybrid zones, had a length of 280 mm measured from the top of the metal component towards the centre of the roof bar. While testing, the lower composite part of the specimen was clamped and fixed with the lower plate. On the upper side, only the wings of the omega shaped metal part were clamped. Due to the lever where the external force is acting, the interface is loaded with an axial force as well as with a momentum. The whole test rig and its working principle is shown in Fig. 8.

The tests were carried out on a ZwickRoell (Germany) Z100 universal testing machine at standard conditions. The upper plate of the testing machine was used to move the tappet with a constant speed of 10 mm/min downwards. The maximum resulting force was taken as quality parameter for the interface.

3. Results and discussion

3.1. Flow-speed-control

The aim of the FSC is to produce high quality parts, reproducible at every shot. Especially the flow front velocity has a strong impact onto the part quality as it is strongly linked to the void content. This is especially important for the OSH-RTM process as voids can migrate into the interface area where they act as crack initiation points. Constant flow front velocity on an optimised level guaranties a low amount of voids. To compare the resulting Darcy velocity while resin injection Fig. 9 shows the Darcy velocity of two representative experiments during the injection. While the mass flow controlled (MFC) (yellow, lower) curve shows a non-uniform behaviour during mould filling, the FSC (blue, upper) curve shows a constant Darcy velocity as soon as the second pressure sensor (p2) is reached. The measured Darcy velocity is only valid as soon as p2 is reached by the flow front as the pressure gradient is always calculated with the distance between the two sensors. From this point on the FSC can operate.

The needed information for the resin viscosity is interpolated due to the viscosity surface described in the viscosity measurement section.

- As resin temperature, the mean value of the thermocouples integrated in the pressure sensors was taken and
- as time value for the viscosity evaluation, the mean between the flow front arrival times at the pressure sensor one (p1) and p2 was taken.

Due to the integrated PI controller, a slight overshoot can be seen at the switchover from the MFCed to the FSCed injection. Furthermore, it has to be stated that only the flow velocity between the two pressure sensors is controlled. Due to the symmetric geometry of the mould, a constant flow front velocity can be expected. However, this drawback can be reduced by adding further pressure sensors along the flow path and using only the latest two for the FSC. In addition, the FSC is very sensitive to batch variations of the preform. As the experiments were carried out close to the limits of the injection unit, the change
of the batch of the CF braid led to one out of three experiments to not reach the target Darcy constantly.

3.2. Interface quality

The influence of the manufacturing procedure (i.e. MFCed and FSCed injection) on the interface quality of the hybrid part, was tested at the interface. For this reason, twelve specimen were tested. One of the MFCed manufactured specimen broke at 4402 N maximum load while the mean value of the other five specimens was 11697 ± 1032 N. Therefore, this value was excluded from further evaluations as it was defined as outlier. In addition, the measurements of the two specimens produced with FSC, where the target velocity was not reached were excluded from data post-processing. Fig. 10 shows mean values of the maximum force measured during the interface characterisation of specimens manufactured by MFC and FSC together with a confidence interval of 95%. The mean value of the FSC is 13172 ± 765 N that is nearly 1.5 kN higher than the average value measured at the MFC specimen, which is stated above.

To identify if the mean value of the FSC specimens is significantly higher than the one of the MFC specimens a hypothesis test was carried out. The result of a 2-sample t-test, performed with the measurements of the four specimens manufactured with FSC and five specimens manufactured with MFC is a p-value of 0.025. For the test, an α level of 0.05 was chosen. As the p-value is lower than the α level, the test reveals that the mean of the maximum force applicable to the interface of the FSC manufactured parts is significantly higher than that applicable to the MFC manufactured parts. Furthermore, one can be 95% confident that the FSC manufactured parts can reach at least a 310 N higher applicable force.

The same test was carried out including the specimens where the target Darcy velocity was not reached constantly. The result shifted the p-value to 0.051. However, this value is still close to the α level of 0.05. The increase can be explained by the drop of the mean value of the FSC produced parts to 12842 ± 1018 N. The reason can be found in the non-constant flow velocity during the whole filling time.

In addition, it has to be stated that the sample size is quite small. This can result in not normally distributed data points and can lead to inaccurate p-values. Nevertheless, the sample size is sufficient to detect a difference between the means of the measurements and therefore it can be stated that the FSC shows a promising alternative to MFC in terms to improve the interface strength of OSH-RTM parts.

4. Conclusion

The work shown in this paper presents an alternative injection strategy for the OSH-RTM process. Darcy’s law is used to evaluate and control the flow velocity during the filling process at an optimised level. Therefore, a series of preliminary tests needed to be carried out including viscosity characterisation of the reacting resin system, capillary rise experiments at the preform to find the ideal flow front velocity and permeability characterisation of the preform. The classical 2D in-plane permeability measurements did not lead to permeability values applicable for the FSC. Several reasons can be found: On the one hand, the 2D in-plane permeability measurements are carried out in a dry preform while Darcy’s law is applied to the FSC in a completely wetted textile. On the other hand, the handling aspect needs to be taken into account. While the preform of the roof bar was built by covering the foam core with the CF braid, the permeability measurement preform was built by opening up the braided sleeve and then placing it in a flat environment. Furthermore, the geometry of the roof bar itself as well as the injection strategy might influence the two permeability measurement strategies. Studies have shown that the measured permeability of the same material can change by more than one magnitude dependent on the measurement procedure [51]. Therefore, it can be inferred that, if possible, the permeability measurements used for FSC should be performed in a process near environment.

It can be concluded that Darcy’s law in combination with pressure sensors can be used to control the flow velocity inside the mould during resin injection. Furthermore, it can be stated that the FSCed injection strategy is able to improve the interface strength of OSH-RTM parts compared to an MFCed injection.
Finally, it has to be pointed out that the shown injection procedure can lead to a more stable process as it controls the flow speed inside the mould. The measured values can also be used to detect large differences between the manufactured parts while resin injection. For instance, the combination of injected mass flow and measured Darcy velocity can be used to detect race tracking. This enables the manufacturer to rise the part quality while reducing rejected parts.

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