MANUFACTURING, PROCESS SIMULATION AND MECHANICAL TESTS OF A THICK COMPONENT PRODUCED BY COMPRESSION-RTM PROCESS

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Abstract

Advanced composite materials have been attracting the interest of many automotive companies for producing light and high-performance components. Out-Of-Autoclave methods have been recognized as the most promising processes to produce CFRP components. One of the most interesting process is Compression-RTM. The aim of this research is to demonstrate the validity of the C-RTM process for producing parts with a thickness greater than 10 mm. The authors fabricated a preform made by 17 layers of PAN based carbon fibre 800 g/m^2 and 600 g/m^2 twill fabric and injected more than 700 g of matrix. Process simulations have been run to test different boundary conditions without producing physical prototypes. Flexural tests have been performed to compare the behavior of thick parts produced by C-RTM with the ones produced by prepreg and autoclave. The results show comparable strength values between the samples tested. DSC, void content and ultrasound measurements have been investigated to fully understand the physical properties of the finished component. Moreover, cycle times has been analysed to demonstrate the effectiveness of the method both from process and product performances points of view. Results show that thanks to the use of C-RTM the manufacturing time decreases by 68% with respect to the autoclave processes.

1. Introduction

Advanced composite materials, especially those based on carbon fiber, have recently attracted the interest of many companies involved in different fields [1]. Light and high-performance components are not only limited to the racing and aerospace sector. The use of composite materials has gained the interest of construction, electronic and infrastructure industries for the production of lightweight components [2]. This is forcing the research in finding new production methods that allow to decrease manufacturing costs and times. The costs and the times related to the traditional autoclave manufacturing process are not suitable for a high volume productions or for low cost components [3]. OOA (Out-Of-Autoclave) methods have been recognized as the most promising processes to produce CFRP (Carbon Fiber Reinforced Polymer) components in a cost-effective way. In particular, technologies based on dry fiber have demonstrated high repeatability, high automation grade and very remarkable performances. One of the most interesting process is C-RTM (Compression Resin Transfer Molding) [4]. Differently from the RTM, where the resin is forced to flow into the dry preform, in the C-RTM, at first the mold cavity is left partially open and then, resin and hardener are mixed and injected into the mold gap at low cavity pressure. After that, the mold is closed completely, and the preform is totally impregnated and cured before demolding of the part [5]. Recently, this process has been gaining the attention of scientific and industrial community. Important players on composites manufacturing such as Dieffenbacher [6] and Krauss Maffei [7] are providing automated solutions for the C-RTM process. At the same time, much research in recent years has focused on this kind of manufacturing process. Baskaran et al. [8] demonstrated that the C-RTM process leads to lower total cost than RTM and HP-RTM in the production of an automotive roof. Simaceck et al. [9] studied the role of material and process parameters in C-RTM, formulating a bi-dimensional numerical model. Then, they validated this manufacturing process and its numerical simulation by producing a pillar for the automotive sector. Other researches aim to better understand the effect of process parameters on the processability and performances of final product. Baskaran et al. [10] defined laws that correlate process parameters with the amount of resin that penetrates in the preform during the injection stage. Merotte et al. [11] investigated the trough thickness fiber deformation of the preform occurred during the compression stage, developing a model to forecast the compaction behavior of the preform and describing the penetration of the resin in the thickness direction. Mamoune et al. [12] proposed an optimization process for the manufacturing of a composite plate with an imposed final height, by meeting the constraint on the maximum closing force of the mold and reducing the mold filling time. Numerical simulations have been recognized as a useful tool for supporting the optimization of infusion and RTM manufacturing processes [10]. Dedicated models and commercial softwares have been used to simulate the impregnation process of the reinforcements. These tools are based on the Darcy's law describing the flow of a fluid through a porous medium. In the scientific literature, different researches exploited virtual simulations for evaluating the behaviour of liquid composite manufacturing processes. For instance, in [13] and [14] numerical models for analysing the resin flow during the RTM process are presented. Poodts et al. [15] used the dedicated software PAM-RTM™ by ESI Group to investigate the filling stage of RTM process for producing a thick CFRP component. Similarly, Oliveira et al. [16] exploited the functionality of PAM-RTM™ to investigate the infiltration of a CaCO3 filled resin in fibrous porous media. For what concerns the Compression-RTM process, Shojaei [17] developed a mathematical model to predict the three-dimensional flow of the matrix during the filling stage of the C-RTM. Moreover, Keller et al. [18] defined a numerical model of rheo-kinetics, flow and heat transfer for optimising the process parameters and ensure complete impregnation without sacrificing cycle time. Furthermore, some researchers prefer to use PAM-RTM™ software for simulating all the stage of the C-RTM process. For instance, Baskaran et al. [8] exploited numerical simulations based on this software to estimate the costs of RTM, HP-RTM and C-RTM. Similarly, Marquette et al. [19] demonstrated the validity of the new Inter-Penetrating Meshes technology presents in PAM-RTM™ in reproducing the compression stage of the C-RTM process. However, what emerges from the literature review is a lack of demonstration that the C-RTM process can be a valid method for producing thick components such as anti intrusion panels for the automotive industry. For this reason, the aim of this article is to demonstrate the feasibility of the manufacturing of a 12mm thick CFRP component by C-RTM. The filling process has been optimised by means of numerical simulations with PAM-RTM™ software. Mechanical flexural tests have been conducted to compare the behavior of the thick part produced by C-RTM with the ones produced by prepreg with a similar matrix system and autoclave. DSC (Differential Scanning Calorimetry) and void content measurements have been performed to fully understand the curing behavior and the matrix amount of the optimal component. Furthermore, thanks to the collaboration with an Italian leading company on composite manufacturing, cycle times has been analyzed to demonstrate the effectiveness of the method for mass production.

2. Manufacturing

2.1. Materials and tools

The thick part to be produced in C-RTM was a flat panel with the following dimensions: width = 282 mm and length = 382 mm. In order to produce a component with a relevant thickness, the laminate was made up of 17 layers of PAN based carbon fiber. The layers were composed of:

- 600 g/m^2 2x2 twill fabric;
- 800 g/m^2 2x2 twill fabric.

The stack sequence is composed by the alternation of these two fabrics: 9 layers of 800 g/m^2 and 8 layers of 600 g/m^2. In this manner, the total areal weight was 12 kg/m^2 and a theoretical thickness of 12 mm. The resin to be injected was the HEXION Epikote Resin TRAC 06150 and the hardener was the

HEXION Curing Agent TRAC 06150. This is a low viscous matrix system designed for RTM applications that allows rapid cycle time. The complete polymerization can occur in 5 minutes at 120°C. Moreover, this high-performance matrix system has a Tg of 124 °C and a tensile strength of 85 MPa.
The mould used in this research is made of Aluminium 7075. It is composed of two parts: mould and movable countermold. In order to maintain the countermold partially open, an inflatable seal made of silicon rubber with a specific profile was used. Thanks to this inflatable seal it was possible to easily manage the gap between the preform and the countermold. Indeed, increasing the inflation pressure results in lifting the countermold. Exploiting a pressure regulator, a fine tuning of the injection gap could be achieved. A powder binder has been applied onto the reinforcement in order to avoid fibre distortion during the injection and the compression stage. Moreover, the presence of the binder allows to reduce the amount of the resin that flows into the preform during the preliminary injection stage. This binder (HEXION Epikote Resin TRAC 06720) was applied at a concentration of 10 g/m^2 and the preform was heated at 140 °C for 60 seconds to allow a significant cure of the binder. The mold was cleaned and released using specific solvents to facilitate the demolding phase.

2.2. Process parameters

The process variables that can be considered as more impacting on the C-RTM process are [10]: (I) gap thickness, (II) injection pressure, (III) injection flow and (IV) resin temperature. The gap thickness was controlled, as mentioned above, by inflating the inflatable seal. The other parameters are directly managed by the matrix mixing machine. It allowed the fine regulation of the temperature of resin and hardener, thus it was possible to keep under control the viscosity of the matrix. Coupling experimental tests and virtual simulations, it was possible to find the best configuration of process parameters for producing components with low resin and void content and with acceptable surface quality. Table 1 shows the value of the best configuration of process parameters.

Table 1. Best configuration of process parameters.

Process parameters	Value
Gap thickness	6,1 mm
Injection flow	440 cm^3/min
Injection pressure	6 bar
Resin temperature	70 °C

2.3. Production process

To produce a component with a matrix mass fraction of 36 %, 725 g of matrix was injected into the preform. However, after several tests, a squezze out of 5 % and a resin excess of 6 % was considered and thus 800 g of the matrix was injected. In this manner, a component with 36 % of matrix mass fraction was produced. The metallic mold with the cured preform were placed in a press heated by hot plates. Vacuum has been applied to the mold cavity. The pressure inside the mold assumed a value of 0,3 bar. All the system was pre-heated at 80 °C in order to avoid thermal shock of the matrix during the injection followed by unexpected increase of the viscosity. The temperature was monitored by the means of thermocouples. In this way, when the temperature inside the mold was homogenous, the matrix was injected. Once the injection phase ended, the seal was deflated. Monitoring the loss of pressure from the seal it was possible to manage the closing velocity of the mould and, thus, the impregnation phase. However, in order to assure mold closure and preform compaction, a clamping force of 25 kN was applied by means of the press. The temperature of the hot plates was increased until reach 140 °C. After several tests at these conditions, it was possible to define as curing time 13 min. At the end of the curing process, the component was demoulded at high temperature in order to avoid mould cooling that can lead to thermal deformations of the carbon fiber component.

3. Process simulation

The design and optimisation of the C-RTM process were conducted through the use of numerical simulation. Numerical simulation is a powerful tool able to reproduce the real behaviour of a product or manufacturing process in a computational environment. In this paper, PAM-RTM® software tool was exploited to investigate the filling process. A proper characterisation of the permeability tensor of the laminate plays a crucial role to achieve a reliable simulation of the filling process [20]. This characterisation is critical since most of the techniques are developed for thin parts, where the through plane permeability (K3) is neglectable. For this reason, this characterization was entrusted to an external laboratory specialized in this kind of analysis. Due to the confidentiality of the data, the achieved permeability values can not be shown. The first step of the simulation process was the creation of a 2D geometry with the relative mesh. Triangular elements were chosen for meshing the component. The stacking sequence of the part (described in section 2.1) was defined and used to extrude the planar mesh into 3D elements. A grid independency check was performed and it was found that extending the cells count above 298700 does not significantly influence final results. Fig. 1 shows the achieved mesh. The boundary conditions considered in this analysis was defined according to the process description provided in section 2.3.

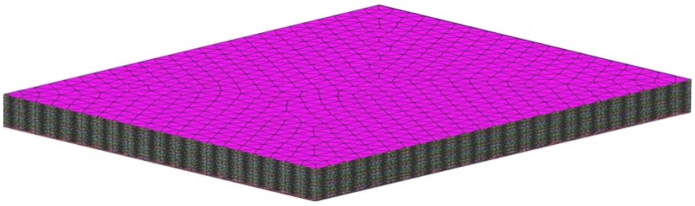
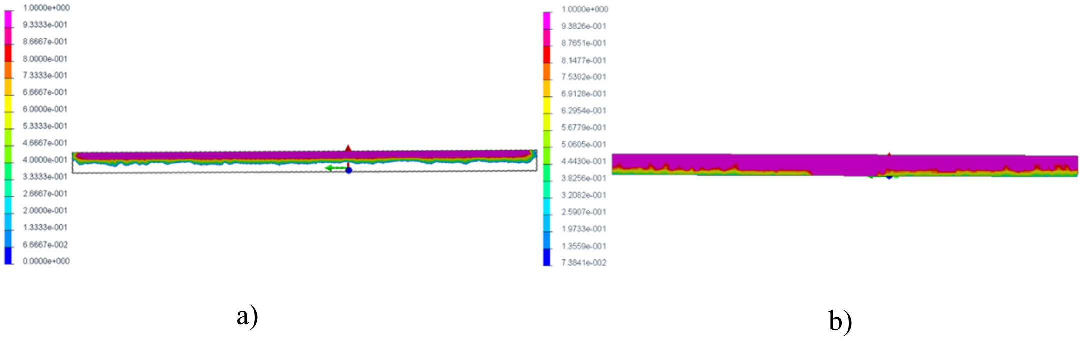


Figure 1. Mesh of the investigated thick component.

3.1. Simulation results

The filling process was analised to define the best values for the process parameters. Simulation results of greater interest for the purpose of this analysis are: pressure field during the infusion, filling factor and filling time. The filling factor (in Fig. 2 is shown the filling factor of the best configuration) is a dimensionless parameter, varying from 0 to 1, that indicates the filling degree in each point of the domain.



a) b)

Figure 2. Filling factor of the best configuration at: a) frame 5,1 sec; b) frame 225,2 sec.

The filling time (in Fig. 3 is shown the filling time of the best configuration) represents the time of filling of the mold, i.e. the time required to impregnate completely the preform.

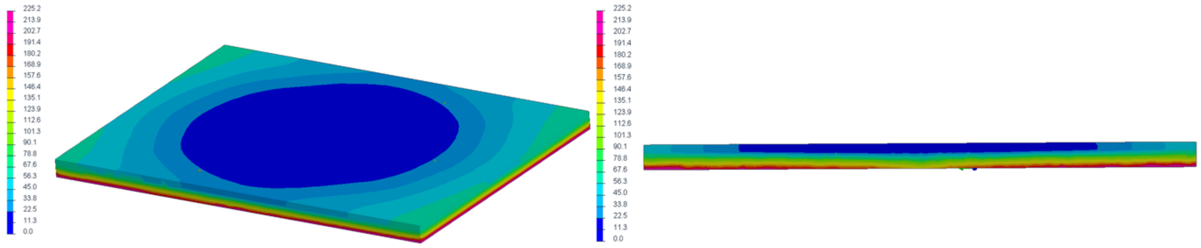


Figure 3. The filling time of the best configuration.

To compare the simulation results with the experimental ones, data provided by the software PAM-RTM was exported and processed. The results showed a good correspondence between numerical simulation and experimental test, with a deviation less than 5%.

4. Experimental test

In order to assess the performances of the process and component, non destructive and destructive tests was performed. In particular, four test procedures were used: ultrasound, resin digestion, DSC and flexural test. A prepreg + autoclave thick component was also produced for a comparative analysis. The reinforcements and the stacking sequence were the same of the dry preform while the matrix system was a structural epoxy resin with a Tg of 130 °C. The manufacturing of this part required several intermediate compaction processes by using vacuum. This sample has been used as a reference for the ultrasound analysis of the components produced by the C-RTM process. It presented a matrix mass fraction of 38 % and the void content, conducing the test in accordance with the ASTM D3171, was 1,9 %. For the ultrasound analysis, Olympus epoch 1000i instrument with v204-rm contact transducer was exploited. The a-scan pulse-echo method was used. The reference standard was set to give an end signal of 80 % when scanning a similar component produced with the traditional process of prepreg and autoclave. The samples that led to a value lower than 90 % of the reference end signal were discarded and change at the process parameters were made accordingly to the numerical simulations. Then, in order to quantify the voids quantity, the ASTM D3171 was used in those components that presented a result of the ultrasound scanning higher than 90 %. The followed procedure was the procedure B: matrix digestion using sulfuric acid/hydrogen peroxide. The result of the optimal component showed a remarkable value of void quantity of 1,5 %. On the same way, DSC analysis was carried out in those samples with ultrasound signal value higher than 90 %. DSC measurements were carried out on a Mettler Toledo DSC1 Star System. The samples were heated from ambient temperature to 220 °C with a heating rate of 20 °C/min. This kind of analysis was used to evaluate the curing time and the curing behaviour inside the mold. More than a sample was taken from the components in order to verify the curing state in different parts, from the inner to the outer layer. In Fig. 4, the DSC analysis of the inner layer of the optimal component, that can be considered as the most critical part because its distance form the hot platens, is reported.

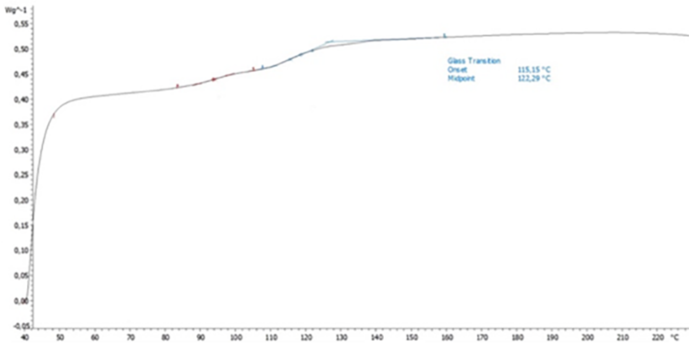
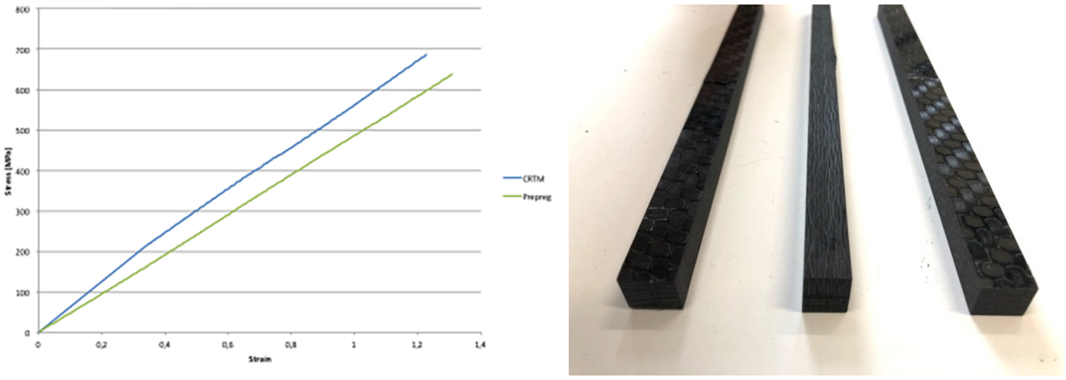


Figure 4. DSC report of the inner layer of the optimal component.

The results of the DSC analysis showed an onset Tg of 115 °C and a midpoint Tg of 122 °C, in line with the data of the technical datasheet of the matrix. The reference sample and the optimal component produced by C-RTM was also tested accordingly to the ASTM D7264. The flexural test provides good information about the mechanical behaviour of the analised components, this because there are combined effects of material's basic tensile, compressive and shear properties. Due to the high thickness of the component, a span-to-thickness ratio of 16:1 was chosen. Thus, the specimen (shown in Fig. 5b) dimensions are: 12x13x192 mm.



a) b)

Figure 5. a) Results of the flexural test of C-RTM and prepreg components, b) C-RTM specimen used for the flexural test.

In Fig. 5, results of the flexural tests of C-RTM and prepreg components are reported. The values represent the average results of the 5 specimens tested. The component produced by C-RTM present higher performances in terms of the maximum stress and modulus. The C-RTM component reports a maximum stress 11% and a modulus 14 % higher than the prepreg one.

5. Cycle time analysis

One of the most important advantage of the C-RTM is its rapidity in the injection and in the impregnation through thickness as well as the curing cycle of the matrix system. In order to demonstrate this, a comparative analysis of the cycle time for manufacturing the thick component by C-RTM and by autoclave was conducted. As shown in Table 2, the total cycle time concerning the C-RTM process was about 4,5 times less than the prepreg + autoclave. Remarkable reductions can be noted for the pre-curing phases (790 sec Vs. 1170 sec) as well as for the curing phase (780 sec VS. 6000 sec).

Table 2. Time breakdown of the C-RTM and autoclave processes.

	C-RTM [sec]	*Prepreg+Autoclave [sec]*
Layup (with binder only for C-RTM)	350	270
Preform curing	200	---
Compactions and vacuum bagging	---	900
Molding (injection+mold closure)	240	---
Curing (with cooling only for prepreg)	780	6000
Demolding	150	210
TOTAL	*1720*	*7380*

6. Conclusion

The presented paper reported the case of a thick CFRP component which was produced using the Compression-RTM method. Numerical simulations were exploited to achieve the configuration of the process parameters that allow to guarantee the efficient filling of the mould avoiding dry zones and minimizing the cycle time. Results of simulations (performed with PAM-RTM) were compared to experimental data. It was noted a good agreement between virtual and real data: the deviation was less than 5 %. Different tests (ultrasound, resin digestion, DSC and flexural test), performed both on components produced by C-RTM and with the Prepreg and Autoclave method, were carried-out. The results show that, through the C-RTM method, it is possible to achieve components with enhanced mechanical performance and higher quality than those produced by Prepreg and Autoclave. Moreover, the total cycle time to produce a part by C-RTM is about 4,5 times less than the Prepreg and Autoclave. Future works will aim to produce more complex products that can be used in real market applications.

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References

[1] M. Holmes. Carbon composites continue to find new markets. *Reinf. Plast.*, 61: 36–40, 2017. doi:10.1016/j.repl.2016.12.060.

[2] V. Mathes. The composites industry: plenty of opportunities in heterogeneous market. *Reinf. Plast.*, 62: 44–51, 2018. doi:10.1016/J.REPL.2017.05.002.

[3] S. Rao, T.G.A. Simha, K.P. Rao, and G.V. V. Ravikumar. Carbon Composites Are Becoming Competitive And Cost Effective. *Infosys Website*, 2–3, 2015.

[4] S. Bickerton, and P.A. Kelly. 11 - Compression resin transfer moulding (CRTM) in polymer matrix composites BT - Manufacturing Techniques for Polymer Matrix Composites (PMCs). in: Woodhead Publ. Ser. Compos. Sci. Eng. Woodhead Publishing, 2012: pp. 348–380. doi:https://doi.org/10.1533/9780857096258.3.348.

[5] P. Bhat, J. Merotte, P. Simacek, and S.G. Advani. Process analysis of compression resin transfer molding. *Compos. Part A Appl. Sci. Manuf.*, 40: 431–441, 2009. doi:10.1016/j.compositesa.2009.01.006.

[6] Lightweight Strategy for the Automotive Industry - Wet Molding Method - An Economical Alternative to Mass Production of CFRP Components Carbon for Large Scale Production. 2015. https://www.dieffenbacher.de/upload/teaser/Flyer/PR/PressesAndMoreComposites2015_EN-web.pdf.

[7] E. Polytechnique, E. Montpetit, and S. Centre-ville. Compression Resin Transfer Molding (C-RTM). 17: 1998. https://www.kraussmaffei.com/rpm-en/compression-resin-transfer-molding-c-rtm.html.

[8] M. Baskaran, M. Sarrionandia, J. Aurrekoetxea, J. Acosta, U. Argarate, and D. Chico. Manufacturing cost comparison of RTM, HP-RTM and CRTM for an automotive roof. *Proceedings of the 16th European Conference on Composite Materials ECCM16*, *Seville, Spain*, 2014.

[9] P. Simacek, S.G. Advani, and S.A. Iobst. Modeling flow in compression resin transfer molding for manufacturing of complex lightweight high-performance automotive parts. *J. Compos. Mater.*, 42: 2523–2545, 2008. doi:10.1177/0021998308096320.

[10] M. Baskaran, L. Aretxabaleta, M. Mateos, and J. Aurrekoetxea. Simulation and experimental validation of the effect of material and processing parameters on the injection stage of compression resin transfer molding. *Polym. Compos.*, 2017. doi:10.1002/pc.24514.

[11] J. Merotte, P. Simacek, and S.G. Advani. Resin flow analysis with fiber preform deformation in through thickness direction during Compression Resin Transfer Molding. *Compos. Part A Appl. Sci. Manuf.*, 41: 881–887, 2010. doi:10.1016/j.compositesa.2010.03.001.

[12] A. Mamoune, A. Saouab, T. Ouahbi, and C.H. Park. Simple models and optimization of compression resin transfer molding process. *J. Reinf. Plast. Compos.*, 30: 1629–1648, 2011. doi:10.1177/0731684411421539.

[13] A. Saouab, J. Bréard, P. Lory, B. Gardarein, and G. Bouquet. Injection simulations of thick composite parts manufactured by the RTM process. *Compos. Sci. Technol.*, 61: 445–451, 2001.

[14] M. Deléglise, P. Le Grognec, C. Binetruy, P. Krawczak, and B. Claude. Modeling of high speed RTM injection with highly reactive resin with on-line mixing. *Compos. Part A Appl. Sci. Manuf.*, 42: 1390–1397, 2011. doi:10.1016/j.compositesa.2011.06.002.

[15] E. Poodts, G. Minak, L. Mazzocchetti, and L. Giorgini. Fabrication, process simulation and testing of a thick CFRP component using the RTM process. *Compos. Part B Eng.*, 56: 673–680, 2014. doi:10.1016/j.compositesb.2013.08.088.

[16] I.R. Oliveira, S.C. Amico, J.A. Souza, and A.G.B. de Lima. Numerical Analysis of the Resin Transfer Molding Process via PAM-RTM Software. *Defect Diffus. Forum*, 365: 88–93, 2015. doi:10.4028/www.scientific.net/DDF.365.88.

[17] A. Shojaei. Numerical simulation of three-dimensional flow and analysis of filling process in compression resin transfer moulding. *Compos. Part A Appl. Sci. Manuf.*, 37: 1434–1450, 2006. doi:10.1016/j.compositesa.2005.06.021.

[18] A. Keller, C. Dransfeld, K. Masania, and A. Northwestern. Numerical Modelling Of Flow And Heat Transfer For The Compression Rtm Process With A Fast-Cure Epoxy. *Proceedings of the 17th European Conference on Composite Materials ECCM-17*, *Munich, Germany*, 2016.

[19] P. Marquette, A. Dereims, T. Ogawa, and M. Kobayashi. Numerical Methods for 3D Compressive Rtm. *Proceedings of the 17th European Conference on Composite Materials ECCM17*, *Munich, Germany*, 2016.

[20] N. Vernet, E. Ruiz, S. Advani, J.B. Alms, M. Aubert, M. Barburski, B. Barari, J.M. Beraud, D.C. Berg, N. Correia, M. Danzi, T. Delavière, M. Dickert, C. Di Fratta, A. Endruweit, P. Ermanni, G. Francucci, J.A. Garcia, A. George, C. Hahn, F. Klunker, S. V. Lomov, A. Long, B. Louis, J. Maldonado, R. Meier, V. Michaud, H. Perrin, K. Pillai, E. Rodriguez, F. Trochu, S. Verheyden, M. Weitgrefe, W. Xiong, S. Zaremba, and G. Ziegmann. Experimental determination of the permeability of engineering textiles: Benchmark II. *Compos. Part A Appl. Sci. Manuf.*, 61: 172–184, 2014. doi:10.1016/j.compositesa.2014.02.010.