

IN-MOULD FLOW AND FIBRE ORIENTATION ANALYSIS OF CARBON FIBRE SMC COMPOSITES

Biruk Nega¹, Robert Pierce², Qihan Hu³, Xiaosu Yi¹ and Xiaoling Liu¹

¹ Department of Mechanical, Materials, and Manufacturing Engineering, University of Nottingham Ningbo China, Ningbo, China

² DTU Wind and Energy Systems, Technical University of Denmark, Roskilde, Denmark ³ Institute for Materials Discovery, University College London, London, UK

Keywords: Sheet Moulding Compound (SMC), Automated preforming, Fibre orientation, Charge flow

ABSTRACT

In this study, flow-induced fibre reorientation in carbon fibre SMCs after compression moulding has been investigated experimentally using an image-based fibre orientation analysis technique. The effect of processing parameters, i.e., charge coverage and initial preferential alignment on fibre reorientation and mechanical performance has been studied. Greater fibre alignment was observed in the direction of greater flow distance, hence plaques with smaller charge coverage exhibited higher flow-induced alignment. Furthermore, panels made from highly aligned longer fibre tows maintained more of the preferential alignment due to the greater resistance of longer fibre tows to flow induced alignment. The measured anisotropy in the tensile properties of coupons taken from two orthogonal directions supported the observations from fibre orientation analysis.

1 INTRODUCTION

Compression moulding of advanced carbon fibre Sheet Moulding Compounds (SMCs), using an automated preforming technique directly from the roving, is becoming an attractive, high volume composite production process due to overall cost and low manufacturing waste advantages. Non-net shaped charges are typically used as significant charge flow can occur during compression moulding, which also allows for manufacturing of parts with ribs and bosses. However, during charge flow the microstructure of the charge changes, introducing local variability, mainly in fibre orientation and other parameters such as volume fraction and void content [1–3]. Understanding of fibre orientation in compression moulded composites and reorientation during forming is thus crucial as it directly influences the mechanical performance and warpage [4, 5]. Simulation tools have been used to predict the evolution of fibre orientation as a function of processing variables, yet the accuracy depends on an assumption of initial fibre orientation and the limitations of the incorporated constitutive models [6].

Optical microscopy has been a widely used experimental technique to infer fibre orientations from the projection of fibre ends at a section of interest. However, its representativeness relies on part homogeneity since the measurement samples are significantly smaller than the actual part. Micro computed X-ray tomography is another common method for fibre orientation characterisation despite limitations in terms of sample size and relative contrast between constituents, particularly in carbon fibre composites [7]. Alternatively, radiography based on x-ray scattering through the composite microstructure has also been used for non-destructive characterisation of fibre orientation [8]. In contrast to micro computed tomography, radiographic methods offer shorter measurement time, greater sensitivity to weak attenuation contrast, and overcome sample size limitations. These measurement methods are often only applicable to cured composite parts. In recent years, image-based non-destructive fibre orientation measurement techniques have been developed that are applicable to wet preforms and cured composites [9–11]. In the current study, one such method that utilizes the optical reflectance behaviour of fibres has been employed to non-destructively characterize the fibre orientation state in the wet initial charge and the compression moulded carbon fibre SMC composites. Fibre orientation tensor components and circular statistics parameters are used to evaluate the effect of initial charge coverage and preferential alignment on flow-induced fibre alignment. Mechanical testing of coupons taken from different directions was used to support the observations from fibre orientation analysis.

2 MATERIALS AND METHODS

Two types of discontinuous fibre composites were prepared from either regularly distributed or highly aligned carbon fibre tows in vinyl ester resin using a Directed Fibre Compounding (DFC) [12] process coupled with compression moulding. Four flat plaques of 3-4 mm thick were manufactured from T700SC-12k carbon fibre tows at 30% nominal volume fraction. The resin used comprises four-part mix, i.e., 1.5 wt.% tert-butylperoxybenzoate hardener, 2 wt.% MgO thickening agent, 4 wt.% zinc stearate (ZnST) in-mix release agent (later replaced by an in-mould agent) and 100 wt.% derakane 782 vinyl ester resin. The schematic configurations of the initial charge coverage for the four plaques are shown in Figure 1. Alternating layers of randomly distributed fibre tows and resin paste were deposited on 250x250 mm area and compacted at room temperature to fill a cavity of 400x400x4 mm. The 50%R charge was prepared by stacking two layers cut from the centre of the compacted 100%R preform while the 25%R was assembled from four quarters stacked orthogonally. All regularly distributed panels were manufactured from 25 mm long fibre tows. The highly aligned charge was prepared from a mixture of 25 and 37.5 mm long fibre tows (in 1:3 ratio) using alignment concentrator mounted with fibre chopping device. Preforms were prepared on 400x400 mm area and cut to 25% coverage and stacked in the same direction. Both types of preforms were thermally B-staged at 50 °C for an hour.





3 CHARACTERISATION

The viscosity of the resin during the preforming stage is critical for the infiltration of fibre tows. Hence, the resin mix viscosity was characterised using a rotational rheometer (Kinexus, NETZSCH) at a shear rate appropriate for the free-falling fibre tows on a resin paste. As can be seen from Figure 2, the measured viscosity was observed to be significantly higher than typically required for carbon fibre SMC preforming [13, 14]. Hence, the powdered release agent was replaced with on-mould release agent to reduce the resin mix viscosity. Although, significant gain was achieved, the steady viscosity of 5 Pa·s is still a concern to properly infiltrate the tows. Hence, an intermediate cold pressing step was introduced for 5 minutes at 10 MPa pressure to improve the infiltration.

Moreover, the cure kinetics of the resin have also been characterized using Differential Scanning Calorimetry (DSC) with isothermal temperature scans (Q20 TA instruments). The curing process under isothermal heating was evaluated for a temperature range of 130 - 150 °C at 10 °C steps. The exothermic curing process shown in Figure 3 was completed in less than 3 minutes for all cases considered. As the curing process can be influenced by non-uniform heating of the mould surface and composite part thickness, the SMC plaques were moulded at a temperature of 150 °C and 10 MPa pressure for 15 minutes to ensure complete curing.



Figure 2: Characterisation of the resin viscosity, with and without in-mix release agent (ZnST).



Figure 3: Isothermal DSC thermogram of the vinyl ester resin at different temperatures.

The fibre orientation distribution of the preforms and moulded plaques were measured using an optical reflectance analysis technique [10, 11]. The experimentally obtained discrete orientation measurements were then fitted using a von Mises distribution (ψ), an approximation of a wrapped normal distribution, to account for the periodicity of the angular measurements, given in Equation 1. The parameters μ and κ represent location and concentration parameters respectively, and $I_0(\kappa)$ is the modified Bessel function of the first kind and order 0. Orientation tensor components were then calculated by forming a dyadic product of a vector representing the orientation of a fibre on a unit sphere and integrating the product with the fitted distribution function over possible angular range as in Equation 2, where Φ and θ correspond to the in-plane and out-of-plane angles respectively.

$$\psi(\Phi;\mu,\kappa) = \frac{exp(\kappa\cos\Phi - \mu))}{2\pi I_0(\kappa)} \tag{1}$$

$$a_{ij} = \int_0^{2\pi} \int_0^{\pi} p_i p_j \psi(\theta, \Phi) \sin\theta d\theta d\Phi$$
⁽²⁾

In addition to the tensor components, circular statistics descriptive measures (kurtosis and skewness) were also used to evaluate the nature and shape of the orientation distribution function before and after moulding. The mechanical performance and anisotropy in the moulded plaques were studied by tensile testing of coupons cut from two orthogonal directions.

3 FIBRE ORIENTATION ANALYSIS RESULTS

3.1 Effect of charge coverage

Fibre orientation analysis results from the front surfaces of the 100%R and 25%R panels are shown in Figure 4 from before and after moulding. The corresponding fibre orientation tensor components and circular statistics parameters are given in Table 1. In the 100%R panel, no charge flow was observed, and fibre tows have mostly preserved their shapes after moulding, except near the corners due to uneven initial charge thickness. Consequently, both tensor components and statistical parameters from Table 1 remained relatively consistent before and after moulding. However, partial charge coverages panels from the regularly distributed tows (25%R and 50%R) showed significant flow-induced radial fibre alignment with the flow direction. The initial charge coverage areas preserved the preferential alignment from the deposition process while greater flow was seen outside, towards the corners as expected. The 50%R panel maintains its initial preferential alignment due to a limited flow distance. However, the 25%R panel was moulded to produce a nearly uniform fibre distribution, showing an 8% a₂₂ tensor component reduction (in the preferential alignment direction), and a 66% reduction in concentration parameter. Overall, all the three panels made from regularly distributed fibre tows showed a moderate concentration parameter and low kurtosis value, implying a nearly standard normal distribution with a slight skewness.



Figure 4: Fibre orientation analysis from the front surfaces of the 100% and 25% charge coverage panels manufactured from regularly distributed fibre tows, before and after moulding.

		100%R		50%R		25%R	
		Before	After	Before	After	Before	After
Tensor	a ₁₁	0.416	0.434	0.421	0.403	0.428	0.474
components	a ₂₂	0.585	0.566	0.579	0.597	0.572	0.526
Statistical parameters	Location parameter, µ	93.7°	89.4°	90.9°	97.6°	100.5°	96.1°
	Concentration parameter, κ	0.346	0.267	0.320	0.411	0.314	0.108
	Skewness, S	0.003	-0.009	0.017	-0.023	-0.020	-0.026
	Kurtosis, K	0.003	0.001	0.055	0.098	0.024	0.044

 Table 1: Fibre orientation tensor components and circular statistics parameters from the front surfaces of panels manufactured from regularly distributed fibre tows, before and after moulding.

3.2 Effect of fibre alignment

Orientation analysis from the front surface of the panel manufactured from highly aligned fibre tows (25% A) is shown in Figure 5 from before and after moulding, along with the corresponding fibre orientation distribution curves. As observed in the 25% R panel, an outward radial flow pattern was observed from the initial charge coverage area. As charge flow is encouraged equally in all directions, a 10% reduction in tensor component in preferential alignment direction (a_{22}) was observed, while the statistical measurement parameters κ and K exhibited greater sensitivity with 38% and 40% reductions respectively. However, the moulded plaque still maintained considerable preferential alignment in the direction 2 as can be seen from the higher a_{22} tensor component, concentration parameter, and kurtosis in Table 2. This is likely due to the greater inertial resistance of longer fibre tows to resist flow-induced reorientation [15]. Low skewness values (close to zero), both before and after moulding, indicate the near symmetric orientation distribution curves around the mean angular orientation of ~90°, which can also be seen from Figure 5.



Figure 5: Fibre orientation analysis from the front surfaces of the 25% charge coverage panel manufactured from highly aligned fibre tows, before and after moulding.

		25%A		
		Before	After	
Tensor	a ₁₁	0.295	0.367	
components	a ₂₂	0.705	0.634	
	Location parameter, µ	89.7°	93.0°	
Statistical	Concentration parameter, κ	0.902	0.557	
parameters	Skewness, S	-0.065	0.016	
	Kurtosis, K	0.153	0.084	

 Table 2: Fibre orientation tensor components and circular statistics parameters from the front surfaces of the panel manufactured from highly aligned fibre tows, before and after moulding.

250/ 4

4 TENSILE TEST RESULTS

Figure 6 and Figure 7 shows the directional tensile stiffness and strength of samples taken from the two orthogonal directions of each panel respectively. Generally, the directional tensile properties showed similar trends with directional tensor components from orientation analysis. The 100%R and 50%R panels showed greater anisotropy, both in stiffness and strength, due to the initial preferential alignment and limited charge flow. However, the 25%R panel, with high in-mould flow, showed near-isotropic tensile properties with 5% and 26% directional difference in stiffness and strength respectively, indicative of a relatively homogeneous fibre distribution. Additionally, the average tensile strength of the 25%R panel also showed a 14% improvement over the panel from 100% coverage. This is likely due to improved impregnation as a result of high in-mould flow [11]. The directional tensile stiffness and strength of the 25%A panel also showed considerable anisotropy, in line with the directional tensor components in Table 2. The tensile stiffness and strength of coupons taken from direction 2 were both seen to be more than three times higher than those of direction 1.



Figure 6: Directional tensile stiffness of panels manufactured from regularly distributed and highly aligned fibre tows.



Figure 7: Directional tensile strength of panels manufactured from regularly distributed and highly aligned fibre tows.

5 CONCLUSIONS

In this study, the effect of initial charge coverage and preferential alignment on the flow-induced fibre alignment and mechanical performance of carbon fibre SMCs has been studied. Greater flow-induced fibre alignment was observed in cases with greater flow distances, hence, lower charge coverage panels showed greater fibre reorientation. Panels with high in-mould flow, 25% coverage, showed improved tensile strength (14%) and lower anisotropy compared with a complete (100%) charge coverage plaque. Moreover, panels moulded from highly aligned longer fibre tows exhibited greater resistance to flow-induced fibre alignment, thus, retaining more of their initial preferential alignment. Consequently, both the tensile stiffness and strength from the alignment direction were observed to be more than three times higher than orthogonal direction.

ACKNOWLEDGEMENTS

The authors would also like to acknowledge the financial support from the Ningbo S&T Bureau under Collaboration Project (Project code: 2017D10033), and the support of the Zhejiang Innovation Team scheme.

REFERENCES

- [1] N. E. J. Olsson, T. S. Lundström, and K. Olofsson, Compression moulding of SMC: Coupling between the flow and the local void contents, *ICCM Int. Conf. Compos. Mater. London*, (2009).
- [2] E. Schmachtenberg, K. Skrodolies, and D. Lippe, Faser/matrix-entmischung während des fliesspressens von SMC, *Zeitschrift Kunststofftechnik/Journal Plast. Technol.*, no. 6, (2005).
- [3] T. H. Le, P. J. J. Dumont, L. Orgéas, D. Favier, L. Salvo, and E. Boller, X-ray phase contrast microtomography for the analysis of the fibrous microstructure of SMC composites, *Compos. Part A Appl. Sci. Manuf.*, vol. 39, no. 1, pp. 91–103, (2008).
- [4] L. T. Harper, T. A. Turner, J. R. B. Martin, and N. A. Warrior, Fiber alignment in directed carbon fiber preforms - A feasibility study, *J. Compos. Mater.*, vol. 43, no. 1, pp. 57–74, (2009).
- [5] S. F. Kite, O. Nixon-Pearson, S. L. Ogin, D. A. Jesson, I. Hamerton, G. Meeks, and A. Sordon, Effect of charge placement on fibre orientation, distortion and failure of a carbon fibre reinforced sheet moulding compound, *ECCM 2018 - 18th Eur. Conf. Compos. Mater.*, no. June, pp. 24–28, (2020).

- [6] C. C. Qian, A. Deshpande, M. Jesri, R. Groves, N. Reynolds, and K. Kendall, A comprehensive assessment of commercial process simulation software for compression moulding of sheet moulding compound, *ESAFORM 2021 - 24th Int. Conf. Mater. Form.*, vol. 02, pp. 1–10, (2021).
- [7] S. C. Garcea, Y. Wang, and P. J. Withers, X-ray computed tomography of polymer composites, *Compos. Sci. Technol.*, vol. 156, pp. 305–319, (2018).
- [8] F. Prade, F. Schaff, S. Senck, P. Meyer, J. Mohr, J. Kastner, and F. Pfeiffer, Nondestructive characterization of fiber orientation in short fiber reinforced polymer composites with X-ray vector radiography, *NDT E Int.*, vol. 86, no. August 2016, pp. 65–72, (2017).
- [9] C. Hopmann, N. Magura, S. Stender, D. Schneider, and K. Fischer, Establishment of an Inline Quality Assurance Method for Fiber Sprayed Preforms.
- [10] R. S. Pierce and X. Liu, Exploiting the optical reflectance behaviour of carbon fibre composites for low-cost inspection and orientations analysis, *J. Reinf. Plast. Compos.*, vol. 39, no. 23–24, pp. 869–879, (2020).
- [11] B. F. Nega, R. S. Pierce, L. Liu, X. Yi, and X. Liu, Fibre alignment and mechanical performance of carbon fibre Sheet Moulding Compounds under different preform compaction, *J. Reinf. Plast. Compos.*, (2022).
- [12] A. D. Evans, C. C. Qian, T. A. Turner, L. T. Harper, and N. A. Warrior, Flow characteristics of carbon fibre moulding compounds, *Compos. Part A Appl. Sci. Manuf.*, vol. 90, pp. 1–12, (2016).
- [13] F. Gortner, A. Schüffler, J. Fischer-schuch, and P. Mitschang, Use of bio-based and renewable materials for sheet molding compounds (SMC) Mechanical properties and susceptibility to fungal decay, *Compos. Part C Open Access*, vol. 7, p. 100242, (2022).
- [14] N. A. W. Z. Xiao, L. T. Harper, A.D. Evans, T.A. Turner, Characterisation of random carbon fibre composites from a directed fibre preforming process: The effects of moulding process and fibre type, vol. 38, no. 3, pp. 755–770, (2007).
- [15] G. Subramanian and D. L. Koch, Inertial effects on fibre motion in simple shear flow, *J. Fluid Mech.*, vol. 535, pp. 383–414, (2005).