PROCESSING EFFECT ON THE DAMAGE TOLERANCE OF RANDOMLY-ORIENTED STRANDS THERMOPLASTIC COMPOSITES

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1 Introduction

Randomly-Oriented Strands (ROS) composites are a bulk moulding compound type of material made of strands of unidirectional pre-impregnated tape [1]. The main advantage of these materials is their great formability compared to continuous fibre composites, which are generally limited to components having small curvature and thickness variations. They also have higher fibre volume fraction than traditional short-fibre composites, thus higher mechanical properties. ROS composites are intended to be used for small intricate compression moulded components having features such as varying wall thickness, tight radii, reinforcing ribs, mould-in holes, etc. However, this process requires very high pressure in order to “push” the material in the cavity of complex parts. This requirement can significantly limit the maximum part size, being dependent on the capacity of the press utilized. It is expected that ROS composites processed at low pressure will result in variable defect density throughout a single part, due to local lack of flow and compaction. These process-induced defects will most likely affect the mechanical properties, thus the importance of understanding the influence of the processing parameters, such as pressure and temperature, on the compaction quality and resulting mechanical properties of these materials.

2 Background

This type of material was first introduced by Feraboli et al. [2–4], who studied carbon/epoxy ROS composites. In their work, flat panels were press-moulded and the strands random distribution was assessed, as well as the tensile, compressive and flexural strength and modulus as a function of the strand length and aspect ratio. Although large variations in the measured mechanical properties was observed, results showed in-plane isotropic behaviour and modulus comparable to that of continuous fibre quasi-isotropic laminates, while strength was significantly lower [2]. Ultrasonic C-scan inspections, verified by the use of targeted microscopic analysis, have been used to characterize regions of signal attenuation in the material, which were attributed to defects such as macro-voids, fibre kinking and resin-rich regions [3]. It was also found that these materials exhibit a particular notch-insensitive behaviour under open-hole tensile and compression loading, which is thought to be associated with internal stress concentrations arising from the heterogeneous sub-structure of the material [4]. Tuttle et al. [5] have also done some work on the mechanical properties characterization of carbon/epoxy ROS material. They used commercially available HexMC® prepregs made out of randomly oriented AS4/8552 carbon/epoxy strands to produce flat panels by compression moulding. The results showed a random strand distribution and fairly constant fibre volume fraction throughout the width of the panels. An increase in tensile modulus was measured in coupons near the edge of the mould and was attributed to an increase in fibre alignment. Han et al. [6] studied the bearing failure behaviour of carbon/PEEK ROS composites co-moulded with fabric layers at various contents and locations in the panel. Results showed that the bearing strength was 30% higher in 100% ROS composites in comparison with 100% continuous fibre fabric composites, while the highest strength was obtained by moulding the ROS with fabric reinforcement in the core of the panel.

A few studies have shown the excellent formability of compression moulded carbon fibre/thermoplastic ROS composites. Han et al. [7] moulded L-curved beams from carbon/PEI ROS composites and measured their interlaminar tension strength. In [8], a
deep flange was moulded from carbon/PEEK ROS composite, where it was shown that the processing pressure can have an influence on the final part quality, mainly influencing the flow of the material in the mould complex cavity. Lastly, a rotorcraft door hinge featuring net-shaped holes was compression moulded out of chopped carbon/PEEK tapes [9]. Bearing tests have shown that the ROS door hinge yield similar strengths to a steel counterpart, although being only 16% of the weight.

3 Low pressure processing

While most of the literature studies on ROS composites address mechanical properties and/or feasibility of moulding complex shaped components, the processing aspects of these materials have not received much attention. A preliminary investigation by the authors on unidirectional continuous fibre carbon/PEEK composites has shown that low pressure processing had negligible effect on the tensile and compressive strength and modulus of the material, but clearly affected its mode I fracture toughness, $G_{IC}$. Similar results were found on ROS carbon/PEEK composites, where low pressure processing had no influence on tensile strength and modulus, although it had an effect on its short-beam strength [10]. These results showed that as opposed to in-plane mechanical properties, interlaminar properties of these materials are sensitive to the processing conditions. For ROS composites, this main finding can be explained by the fact that their failure is mainly due to matrix microcracking and interply interface disbond, and that fibre breakage is minimal [2].

To further understand the relationship between processing and mechanical properties, this paper presents the results of an investigation of the effect of the processing pressure on the damage tolerance of ROS composites. The compaction quality of panels manufactured at high and low pressure was assessed by means of cross-sectional micrographs and ultrasonic C-scan analysis. Drop-weight impact tests were performed on the panels and the damage characteristics and impact properties were compared between the two processing conditions. All characteristics and mechanical properties obtained in this study were also benchmarked against a quasi-isotropic continuous fibre composite.

4 Experimental procedures

4.1 Material and panel fabrication

The material used in this study was a unidirectional carbon/PEEK prepreg, AS-4/TC1200, supplied by TenCate. The prepreg was chopped into 25 mm long, 6.35 mm wide strands. Two flat ROS composite panels, 356 mm × 305 mm, were manufactured in a compression mould made out of a picture frame and two flat plates. The first panel was moulded using the manufacturer’s recommended high pressure cycle, 3.45 MPa [11]. The second panel was moulded at 1.0 MPa. A 32-layer quasi-isotropic continuous fibre panels moulded at a pressure of 3.0 MPa was used to benchmark the mechanical properties of the ROS composites obtained in this study. Table 1 shows the three panels characteristics. The low- and high-pressure processed ROS panel will be identified as LP–D and HP–D, respectively. The continuous fibre panel will be referred to as HP–C.

The moulding process was the following. The prepreg strands were manually placed inside the mould cavity in small batches in order achieve a random in-plane distribution and minimize out-of-plane orientation. A total of 776 g of material was used to reach an average panel thickness of approximately 4.50 mm. Three thermocouples were inserted in the material, at approximately 13 mm from the picture frame. They were used to monitor the material temperature during the moulding cycle. Once the mould was closed, it was placed in a press and a force of 89 kN was applied, corresponding to a pressure of 0.82 MPa. The press platens were then heated to 400 °C. When the material reached 380 °C, the desired moulding pressure was applied and kept constant for the rest of the cycle. The material was held at the processing temperature for 15 min. Once the dwell was done, the mould was cooled down in three stages: with air from the processing temperature to 343 °C, a mixture of air and water from 343 °C to 177 °C, followed by water only from 177 °C to room temperature. The two ROS composite panels and the moulding cycle of the HP–D panel are shown in Fig. 1 and 2, respectively.
4.2 Destructive and non-destructive inspection

Multiple 25 mm wide samples were taken from each panel in order to inspect their microstructure and measure their void content. The samples were polished and observed under optical microscope at 50X. The void content in each sample was measured with the help of image processing software by comparing the total void area to the total area of the sample. Further inspection was performed on the low-pressure processed panel by means of through-thickness ultrasonic C-scan using a 2.25 MHz sensor.

4.3 Drop-weight impact

Drop-weight impact tests were performed according to ASTM D7136 standard [12]. This mechanical test was selected for this work because the resulting damage is mostly interlaminar, which makes it a great candidate for detecting differences in the material due to processing. Another advantage of impact testing with regards to ROS composites is the large sample size. This is important because the repetitive unit-cell of ROS composites is relatively large and is also dependent on the strand geometry.

All tests were performed on an Instron drop-weight impact tower, model 8200. The impactor was hemispherical and 16 mm in diameter. The impact energy was 30 J for all specimens, based on the ASTM standard’s recommended of 6.7 J/mm. An indication of the degree of damage induced by impact loading was obtained by measuring the contact force and the total energy absorbed versus time during the test. All load-time data was obtained directly from the data acquisition system. The dent depth was measured on each sample immediately after impact using a depth gage micrometer having a spherical tip.

5 Results

The results of this study are presented in three sections. First, the effect of the processing pressure on panel quality is assessed with the help of visual observations, cross-sectional micrographs and ultrasonic C-scan analysis. Second, the impact testing results of the three panels are presented, followed by the post-impact damage evaluation.

5.1 Effect of pressure on panel quality

A considerable difference can be observed between the two ROS panels presented in Fig. 1. A white triangle-shaped region can be observed on the LP–D panel (Fig. 1b), which was determined to be PEEK resin on the surface. These regions had a matt and rough surface, in comparison with the other regions of the panel, which were smooth, shiny, and black. A small number of these regions can also be observed on the right side of the HP–D panel (Fig. 1a). The formation of these white regions is explained in the following.

The cooling system of the press platens is such that the coolant flows (in reference to Fig. 1) from right to left in a main conduit and from the center to the top and bottom in secondary conduits. This created an in-plane temperature gradient on the panel, where TC2 < TC1 < TC3 at all time during cooling. Temperature gradients will delay solidification, crystallization, and thermal contraction of the “hot” regions of the part, compared to the “cold” regions. In this case, the maximum measured temperature gradient was 71 °C, recorded at 110 min into the moulding cycle.

At the processing temperature, the material is molten and the applied load is equally distributed, i.e. the pressure can be assumed to be equals the processing pressure at any point \( P = P_{\text{processing}} \). When the cold material solidifies \( T^* < 343 \, ^\circ \text{C} \) for PEEK), its modulus starts developing. Since the hot material is still molten, the cold material carries most of the applied load \( P > P_{\text{processing}} \). When the hot material starts to solidify, the cold material has already started shrinking due to its ongoing crystallization, in addition to its thermal contraction. This will lead to a non-uniform pressure distribution on the panel. Additionally, during the rest of the cooling process, the applied pressure needs to be high enough for the elastic strain to overcome the additional strains in the cold material due to the thermal gradient. Insufficient applied pressure could lead to a zero pressure zone (i.e. a gap between the press platens and the material) in the cold region, which could have consequences on the final part quality. It is thus believe that this condition was not satisfied during moulding of the LP–D panel, resulting in white PEEK resin regions observed on its surface.
The effect of the white PEEK regions on the panel quality was investigated by performing through-thickness ultrasonic C-scan on the LP-D panel. The results are presented in Fig. 3. The panel is represented in five samples, B1–B5, which are directly comparable with the samples in Fig. 1b. A triangle-shaped region of signal attenuation can be observed on the right of the panel. Clear correlation can be made between the white regions on the surface of the panel and the regions of signal attenuation. These differences in density could be explained by a higher number of defects such as void and resin-rich areas, or simply different levels of compaction in the material due to uneven pressure distribution during processing.

Void content analysis was performed on the three panels. Samples were taken along the dashed line, as shown in Fig. 1. Eight, nine and three samples were analysed from the LP-D, HP-D and HP-C panels, respectively. Two micrographs of the LP-D panel are presented in Fig. 4. The sample in Fig. 4a was taken from the leftmost side of the panel, while the sample in Fig. 4b was taken on its rightmost side, exposing the difference between black and white surface regions of the panel. Although it was almost twice as high in the white surface sample (Fig. 4b), the void content was found to be low in both samples, i.e. 0.20 and 0.36%. The average void content measured in the LP-D samples was 0.28 ± 0.05%. Representative micrographs of the HP-D and HP-C panels are presented in Fig. 5. The average void content in the HP-D was 0.09 ± 0.07%. Most of the voids in both discontinuous fibre panels were located in resin-rich areas at strand tips, as depicted in the zoom region of Fig. 4b. Finally, very few voids (< 0.05%) were observed in the HP-C panel.

5.2 Impact tests

The impact resistance of the three panels was compared based on three characteristic parameters [13]. First, the incipient damage load, \( F_i \), and its corresponding energy, \( E_i \), recorded at the first discontinuity of the force vs. time curve, below which the material experiences no impact damage. Second, the maximum recorded contact force, \( F_{\text{max}} \), an indication the maximum load that the composite can withstand without undergoing major damage. Lastly, the total energy absorbed by the specimen during the impact, \( E_a \), which is the difference between the applied impact energy and the elastic energy released by the samples, calculated based on the rebound acceleration, force and displacement of the impactor. It is an indication of the degree of damage induced by impact loading, where a perfectly elastic impact event would have \( E_a = 0 \).

The impact load vs. time and energy vs. time curves of representative samples are presented in Figs. 6 and 7. Average results and standard deviations for \( F_i, E_i, F_{\text{max}} \) and \( E_a \) are presented in Table 2. All load curves show two or three discontinuities at the beginning of the impact event, which were neglected in the \( F_i \) measurements as they were attributed to harmonic resonance of the impactor and/or sample [12]. Fig. 6 shows clear evidence of the higher impact performance in the continuous fibre panel. Average \( F_i \) and \( F_{\text{max}} \) in both discontinuous fibre panels were 18.7% and 22.6% lower than those measured in the continuous fibre panel. \( F_i \) and \( F_{\text{max}} \) values were very similar between the discontinuous fibre panels, with differences of 1.3% and 0.4%, respectively. The shorter impact time in the HP-C panel was attributed to its superior stiffness, which was 11.8% higher when compared to the discontinuous fibre panels. Average \( E_i \) energies measured in both discontinuous fibre panels were 25.1% lower than those of the continuous fibre panel. The total absorbed energy, \( E_a \), was the impact parameter most affected by the fibre architecture, where the average value was 42.3% higher in the discontinuous fibre panels. Differences in \( E_i \) and \( E_a \) between the LP-D and HP-D panels were only 2.4% and 5.3%, respectively, in favour of the LP-D panels.

One-way analyses of variance (ANOVA) were performed in order to determine if processing pressure had a significant influence on the impact behaviour of the discontinuous fibre panels, and whether or not the impact results were significantly different between the continuous and discontinuous fibre panels. The analyses were performed on parameters \( F_i, E_i, F_{\text{max}} \) and \( E_a \). Results showed that for both discontinuous fibre panels, the pressure had no influence on any of the impact parameters tested. However, it was found that the fibre architecture, continuous vs. discontinuous, had a significant influence on all impact parameters.
5.3 Post-impact damage evaluation

Visible impact damage on the underside of representative test samples are shown in Fig. 8. The damage on both discontinuous fibre panels was mainly composed of delamination around the strands, cracks, and small amount of fibre failure (Figs. 8a–b). Small cracks were also observed near the dent on the impacted side of the samples. Nonetheless, no clear differences in the failure modes were observed between the LP–D and HP–D panels. The surface damage on the underside of all four HP–C panels (Fig. 8c) was comprised of long delaminations in the 45° surface ply. Apart from the impact dent, no visible damage was observed on the impacted side of the samples.

The average dent depths of the three panels are presented in Table 2. The smallest and largest average dent depths were measured in the HP–C and HP–D panels, respectively. Greater damage length could explain the smaller dents in the HP–C panel, where delamination paths between the layers are prone to larger internal damage. In opposition, the random mesostructure of the discontinuous fibre panels possibly makes it more difficult for cracks to grow, which is reflected by a more localized damage area.

6 Conclusions

The effect of the processing pressure on compaction quality and impact properties of ROS composites was investigated. Two discontinuous fibre ROS panels were moulded at high (3.45 MPa) and low (1.0 MPa) pressure. The internal structure of both ROS panels was very similar, where the difference in average void content was only 0.19%. White PEEK resin and a rougher surface finish were observed in some region of the low-pressure processed panel. This was attributed to the high temperature gradients in the panel during cooling, which lead to a loss of effective pressure between the press platens and the material when processed at low pressure. The white region on the panel was clearly detected via ultrasonic C-scan. However, it was not clear if the difference in void content was solely responsible for these signal attenuations. This will be further investigated in future work.

Impact tests were performed at an energy of 30 J. All impact parameters measured on both ROS composites panels were insensitive to the processing pressure and difference in void content, as verified by ANOVA analyses. The damage modes were also similar for both panels. Thus, for the temperature cycle employed, a pressure of 1.0 MPa was satisfactory to obtain a decent panel quality, with regards to impact resistance.

The fibre architecture had a significant influence on the impact properties of the material; in comparison with a quasi-isotropic continuous fibre laminate, the maximum contact force was 22.6% lower, while total absorbed energy was 42.3% higher in the ROS panels. The fibre architecture also greatly influenced the impact damage modes observed in the panels. Further investigation of the impact damage will be performed in future work by measuring the damage area with the help of ultrasonic C-scan. Cross-section of impacted regions will also be polished and observed under optical microscope, where failure paths will be compared between low- and high-pressure moulded ROS and quasi-isotropic panels.

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References

Table 1. Characteristics of the three panels manufactured and tested in this study.

<table>
<thead>
<tr>
<th>Panel</th>
<th>Configuration</th>
<th>Pressure (MPa)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HP-D</td>
<td>ROS</td>
<td>3.45</td>
<td>4.52</td>
</tr>
<tr>
<td>LP-D</td>
<td>ROS</td>
<td>1.00</td>
<td>4.57</td>
</tr>
<tr>
<td>HP-C</td>
<td>[45/-45/0/90]</td>
<td>3.00</td>
<td>4.34</td>
</tr>
</tbody>
</table>

Fig. 1. ROS panels processed at (a) 3.4 MPa and (b) 1.0 MPa. The press platen’s coolant flows from right to left during cooling. Dots indicate thermocouple location, dashed line indicate area where micrographs were taken.
**Fig. 2.** Pressure cycle and panel temperature measured during moulding at three locations (see Fig. 1) on the HP–D panel.

**Fig. 3.** Through-thickness ultrasonic C-scan of the LP–D panel. Signal attenuation can be seen on the right side of the panel.

**Fig. 4.** Cross-sectional micrographs of the LP–D panel taken at (referring to Fig. 1b) (a) M1. (b) M2.

**Fig. 5.** Cross-sectional micrographs of the two high-pressure processed panels. (a) HP–D. (b) HP–C.
Fig. 6. Typical impact load vs. time curves showing incipient damage load $F_i$ for the three panels tested.

Fig. 7. Typical energy vs. time curves for the three panels tested. The total absorbed energy, $E_a$, is shown for the HP–C panel.

Fig. 8. Visible impact damage on the underside of the test samples. (a) LP–D. (b) HP–D. (c) HP–C.

Table 2. Impact testing results.

<table>
<thead>
<tr>
<th>Panel</th>
<th>$F_i$ (kN)</th>
<th>$E_i$ (J)</th>
<th>$F_{max}$ (kN)</th>
<th>$E_a$ (J)</th>
<th>Dent depth (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HP–D</td>
<td>6.71 ± 0.92</td>
<td>8.37 ± 2.52</td>
<td>9.45 ± 0.48</td>
<td>14.35 ± 1.92</td>
<td>0.90 ± 0.14</td>
</tr>
<tr>
<td>LP–D</td>
<td>6.80 ± 0.86</td>
<td>8.17 ± 2.10</td>
<td>9.41 ± 0.67</td>
<td>13.63 ± 1.20</td>
<td>0.76 ± 0.07</td>
</tr>
<tr>
<td>HP–C</td>
<td>8.31 ± 0.28</td>
<td>11.04 ± 0.85</td>
<td>12.19 ± 0.21</td>
<td>9.83 ± 0.62</td>
<td>0.50 ± 0.02</td>
</tr>
</tbody>
</table>