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Development of a Recycled Compression Moulding Compound for Fast Curing Prepreg Waste

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1. Abstract

Elevated temperature staging of uncured prepreg scraps has been proposed to prevent prepreg scraps from being landfilled. The goal to reuse staged material in compression moulding applications requires that the staged material demonstrates strong shear flow characteristics. A one-dimensional flow compaction experiment was conducted to determine the shear flow of Cycom® EP 2750 at different degrees of cure and viscosities using an instrumented press capable of reproducing diverse moulding conditions. Experimental results were combined with cure and viscosity models to develop a processing map. This was compared to a similar processing map produced by Smith et al. for a prepreg for which the viability of elevated temperature staging as a means of recycling has already been demonstrated. In the initial phase, percolation flow dominated flow behaviour, suggesting poor compression moulding characteristics. By increasing the degree of cure of the recycled material, the compression moulding behaviour of the material was improved, and glass transition development allowed it to be stored at room temperature. Specifically, staging the material to a degree of cure of approximately 35% maximized shear flow and raised the glass transition temperature to approximately 30°C, sufficient to minimize cure advancement at room temperature. In short, the processing parameters for Cycom® EP 2750 suggest the feasibility of elevated temperature staging to recycle uncured scrap Cycom® EP 2750 for compression moulding applications.

2. Introduction

One quarter of prepreg composite materials used in aerospace applications are scrapped before they are cured; either as offcuts, or due to production defects [1]. These scraps, although no longer usable in primary autoclave or compression moulding applications, can be recycled into a room temperature stable recycled prepreg moulding compound (rCMP) through an elevated temperature staging process, as demonstrated by Smith [2]. This process involves staging the prepreg to raise its viscosity, making the material suitable for compression moulding, and raising the glass transition temperature, reducing tack and minimizing the effect that out-life has on cure

advancement. Once staged, the material can be chipped into small pieces and used in place of bulk moulding compounds in elevated temperature compression moulding applications. This novel recycling technique has the potential to reclaim portions of the estimated 50 tons of scrap material currently landfilled [3] by reprocessing it into components that, while not suitable for primary load-bearing structures, still carry the benefits of stiffness and weight reduction otherwise associated with carbon fibre prepregs.

A critical piece of information in determining the suitability of a material as an rCMP ready for compression moulding is to understand the flow compaction behaviour of the staged material. This allows one to assess the magnitude and nature of the flow that will occur during reprocessing. The nature of the flow is characterized by two phenomena: percolation flow, where the resin can flow past the fibre bed relatively uninhibited, and shear flow, where the resin is sufficiently viscous to transport fibres with it as it flows. A good rCMP should exhibit high shear flow magnitude, as this will ensure that the fibre reinforcement is transported to all corners of the mould during processing, minimizing processing defects.

This paper investigates the flow compaction behaviour of Cycom® EP 2750 (herein referred to as EP 2750) [4] that has been a challenge for the proposed recycling method. Already designed for compression moulding, its fast-curing, high viscosity nature means that the processing window to produce a suitable rCMP is smaller than that of comparable autoclave prepreg scraps. Specifically, the range in degree of cure between where the glass transition temperature is above room temperature and where the resin gels, is expected to be less than 10%. Further, the plateau of maximum shear flow reported by Smith must be within that range of cure for EP 2750 to be usable for this form of recycling.

3. Methodology

The experiment presented here is a one-dimensional flow compaction design used by Hubert and Smith to characterize the flow compaction behaviour of prepreg tapes and woven fabrics [2,5].

3.1. Sample Preparation

Samples were cut from scrap EP 2750 of unknown out-time supplied by Bell Textron Inc. As each sample might have had a slightly different initial degree of cure, samples were cut in batches from the same piece of scrap material. Samples were named according to their batch, and the sequential trial number for that test. All samples in Figure 3 are from the same piece of prepreg (Batch 4) and increase in trial number. This was done to account for any variation in flow from batch to batch while still using all available material.

Five levels of staging were tested in addition to an as-received control, with staged viscosity targets of 10 Pa·s, 50 Pa·s, 100 Pa·s, 250 Pa·s and 500 Pa·s. Sample chips measuring 12.7 mm × 12.7 mm (nominally) were placed between two aluminum plates preheated to 140°C and staged in a 140°C oven until the target viscosity was reached, as previously determined through rheological data. Samples were immediately transferred to a room temperature aluminum plate to arrest cure when staging was complete.

Laminates of six plies were then laid up in a $[0]_6$ configuration; equivalent to an approximate thickness of 1 mm. Laminate samples were weighed and the length and width of each ply were measured to determine average pre-processing dimensions (Table 1).

Table 1. Flow compaction sample initial conditions

Target Viscosity (Pa·s)	Staged DOC (%)	Staged Viscosity (Pa·s)	Staged T_g (°C)	Average Sample Mass (mg)	Average Sample Length (mm)	Average Sample Width (mm)
Neat	0	3	−2.6	311.2	12.89	12.72
10	10	10	5.0	309.6	12.58	12.93
50	14	68	22.3	321.3	13.42	13.41
100	34	135	29.1	319.0	13.00	13.20
250	48	282	68.7	315.2	13.29	13.49
500	55	467	73.4	325.6	13.53	13.76

3.2. Experimental Procedure

The experiment was conducted in the instrumented mould that was mounted to an MTS electromechanical testing device equipped with a 5kN load cell (Figures 1a, b).

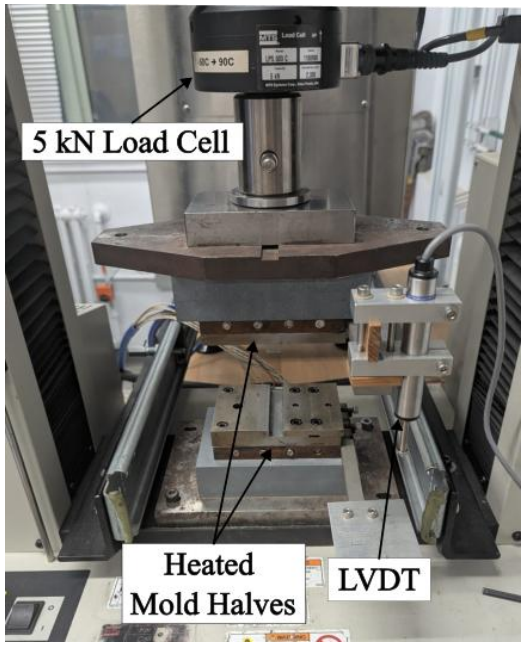

Figure 1a. Instrumented mould mounted in MTS test frame

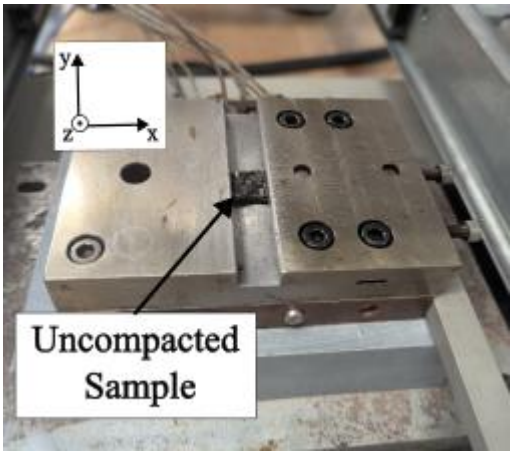

Figure 1b. Inset of mould channel with uncompacted specimen

The experimental procedure described below follows that described by Smith using the same experimental apparatus.

1. The mould was preheated to 175°C, a temperature at which EP 2750 will fully cure in less than one hour and closed until a small load (~10–25N) was registered indicating that the

mould halves were in contact. The Linear Variable Differential Transformer (LVDT; Figure 1a) and MTS crosshead measurements were tared and the mould reopened.

2. The prepared sample was placed in the pre-heated channel of the mould (Figure 1b) and closed until a load of 25 N was registered, indicating that the mould halves were in contact with the sample. The samples were then left to heat-soak for 1 minute to have a consistent temperature through the thickness of the part.
3. The mould was closed at a rate of 0.1 mm/s until a load of 1 kN was registered.
4. The MTS shifted from displacement control to load control and the sample was left to cure under 1kN load and 175°C for 1.25 hours.
5. The heating was turned off and the sample cooled under 1kN load for a further 30–45 minutes until the mould returned to room temperature.
6. The cured sample was cleaned of flashing and reweighed. Any samples with a mass difference of greater than 5% were discarded.

3.3. Microscopy

Following cleaning and reweighing, accepted samples were sectioned along their y-centerline, as per the axes in Figure 1b and potted in preparation for optical microscopy. Following polishing of the potted samples, the resulting cross-sections were photographed under microscope.

The resulting micrographs (Figure 3) were thresholded in ImageJ Fiji using Otsu's method for thresholding to eliminate human bias [6] (Figure 2a). Fibre-rich sections of the sample appear as white areas, whereas resin and voids are black. By tracking the percentage of black vs. white pixels in each vertical column of any image, it was possible to determine the fibre volume fraction at any point in the sample (Figure 2b) and thereby the extent of shear flow. The limit of shear flow was recorded as a fibre volume fraction of less than 20%. This is apparent for the sample at approximately 6 and −6 mm (Figure 2b).

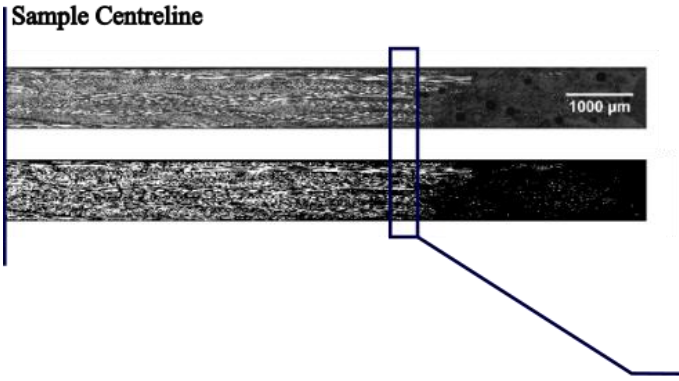

Figure 2a. Micrograph images of raw and thresholded samples.

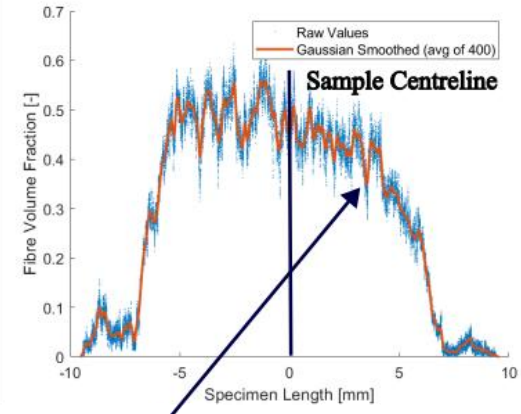

Figure 2b. Fibre volume fraction vs. specimen length from centreline.

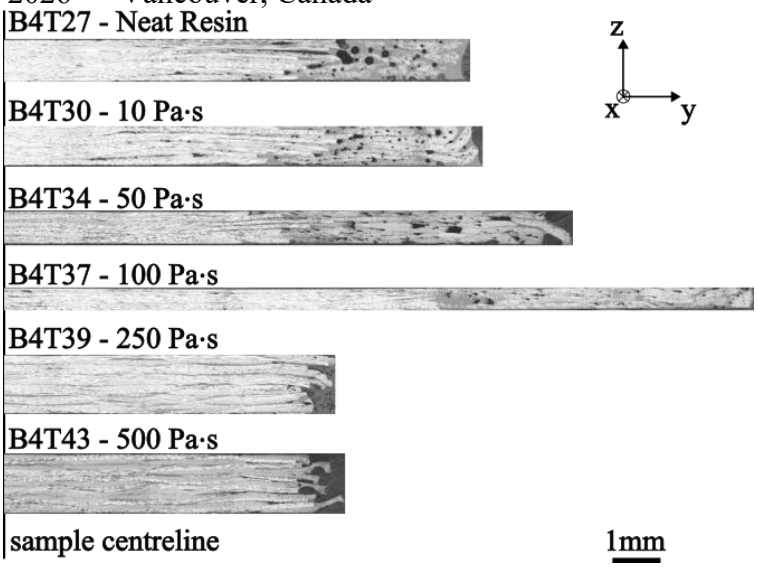

Figure 3. Micrographs of each tested staging level taken from the sample midplane

3.4. Stress and Shear Strain

To determine the stress on the sample as it is compacted, the volume of the uncompacted sample was found using Equation 1:

$$V = \overline{L_0} \cdot \overline{W_0} \cdot h_0' \tag{1}$$

where $\overline{L_0}$ is the average uncompacted sample length, $\overline{W_0}$ the average uncompacted sample width and h_0' the uncompacted thickness of the laminate, taken as the thickness measured at the moment the load cell registered a 25 N load (Step 2 in the procedure). The average lengths and widths are shown in Table 1. The normal stress was then calculated using Equation 2 assuming the average length and width of the samples:

$$\sigma(t) = \frac{F(t)h(t)}{\overline{L_0} \cdot \overline{W_0} \cdot h_0'} \tag{2}$$

Using the thresholded volume fraction data to determine the final length of each sample following compaction, the shear strain in the y-direction was calculated using a one-dimensional form of Green's Strain Tensor (Equation 3). This strain tensor is better suited to large strains like those present in the 50 and 100 Pas samples than engineering strain, due to the quadratic terms [7].

$$e_y^s = \frac{1}{2}\left[\left(1 + \frac{L_f - \overline{L_0}}{\overline{L_0}}\right)^2 - 1\right] \tag{3}$$

4. Results and Discussion

In the as-received and 10 Pas trials, there were significant voids and little reinforcement in the flow area (Figure 3). This suggests that, although there is some shear flow due to reinforcement being pulled along with the resin, percolation flow is still the dominant mechanism. At the other end of the spectrum, the 250 and 500 Pas trials saw negligible flow in the y-direction, and comparatively little compaction (Figure 4). Between the two extremities, though, the 50 Pas and 100 Pas trials resulted in significant shear flow. The thickness of these samples decreased the most (Figure 4), and there was ample shear flow, as expressed by shear strain (Figure 6).

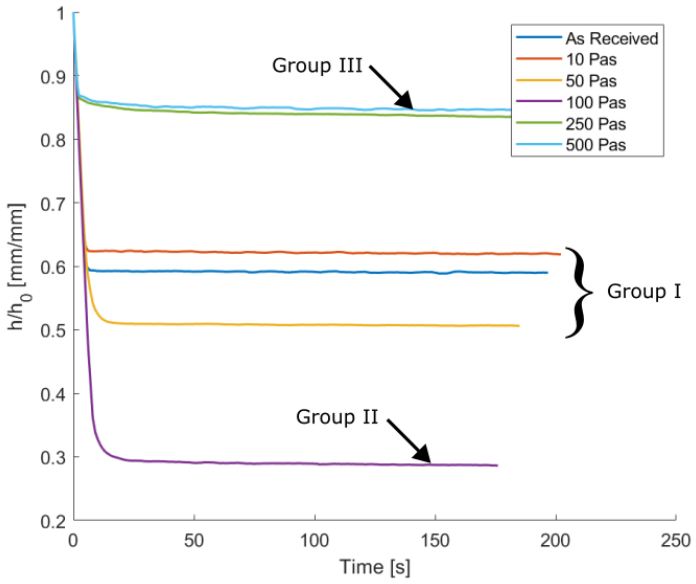

Figure 4. Thickness development during compaction

There was negligible flow in the higher viscosity samples. While validation of the viscosity through correlation with the degree of cure would suggest that the staging process was precise to within ±35 Pa·s of the target viscosity, the experimental process disregarded the heat soak prior to compaction in viscosity development. Samples were staged at 140°C, and modelling would suggest that the post staging viscosity was in the range of the target viscosity. However, the subsequent heat soak at 175°C prior to compaction causes further viscosity development, meaning that the higher viscosity samples likely gelled prior to compaction. We modelled viscosities, including a heat soak of two minutes at 175°C following staging (Table 2).

Table 2. Estimated viscosities at the moment of compaction

Target Viscosity (Pa·s)	Staged Viscosity (Pa·s)	Estimated Viscosity at Compaction (Pa·s)
10	10.5	18.5
50	68.5	173.2
100	135.4	406.5
250	282.5	1175
500	467	2732

Examining normal stress development (Figure 5) provides insight into the different flow compaction mechanisms occurring. There are three distinct groupings. Group I consists of the as-received prepreg, 10 Pas, and 50 Pas samples. The flow compaction curve of this group mimics that reported by Hubert of exponential growth as effective fibre volume fraction increases [8]. The highest maximum stress is observed in Group III. This has an even more dramatic increase in normal stress, suggestive of resin that has gelled before compaction, taking the majority of the compressive load. Finally, Group II is the 100 Pas sample, where the stress development is quasi linear. This suggests that the stress is predominantly increasing along with viscosity development, meaning that the reinforcement is flowing in the y-direction rather than resisting compaction. It appears likely that the 50 Pas sample sits between Groups I and II, as it shows an initial linear increase in stress. During this initial flow, there is sufficient shear that the tows perpendicular to the flow (i.e., tows aligned with the x-axis) are dragged into the flow region. However, the resin is still too thin to draw out the tows aligned with the flow, which are eventually compacted and show the resulting exponential increase in stress.

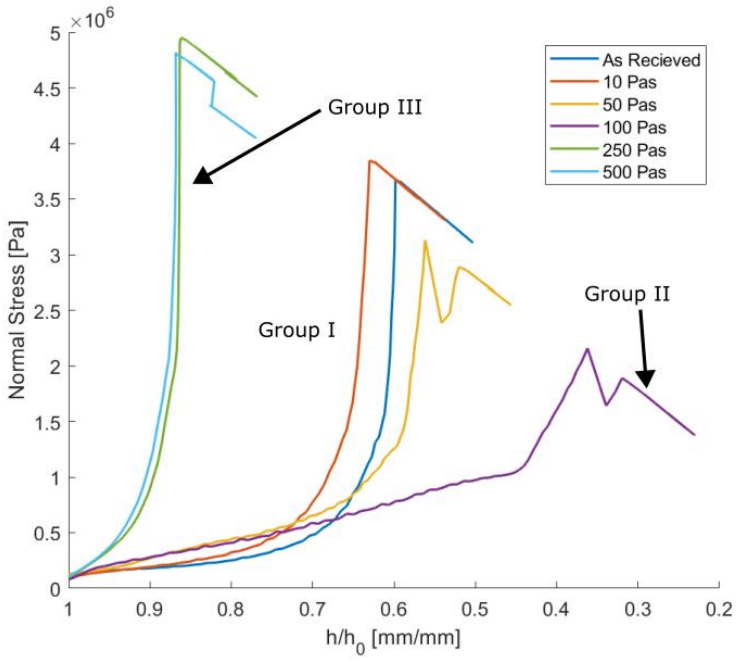

Figure 5. Stress development during compaction vs. thickness development. Note that thickness decreases from left to right.

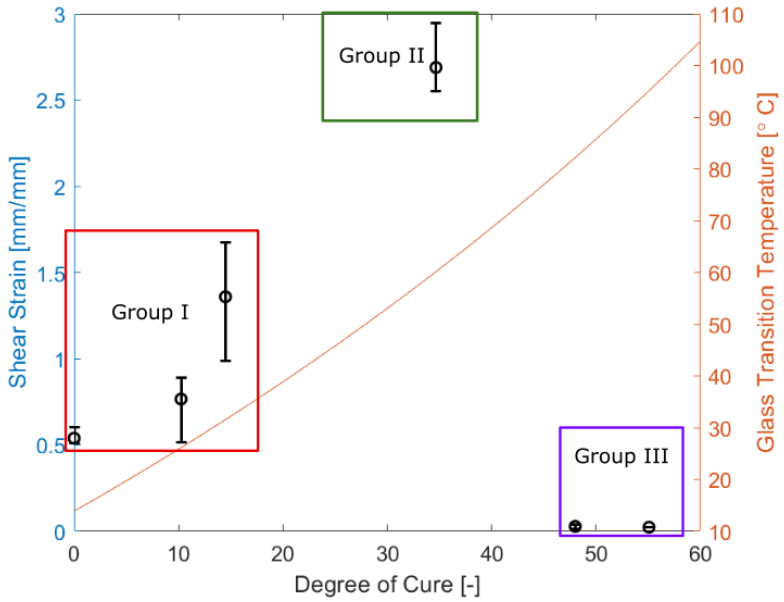

Figure 6. Shear strain and glass transition development of EP 2750 as a function of degree of cure.

There was a clear window of high shear flow around the 50–100 Pas target viscosity samples that demonstrates the feasibility of elevated temperature staging as a method of recycling EP2750. More testing will be required to confirm the true viscosity of the samples when compaction occurs. Further, the glass transition temperature (Table 1, Figure 6) is sufficient to effectively arrest further

cure development at room temperature, which eliminates the need to store the staged samples frozen.

4.1. Comparison to 5276-1

Research conducted by Smith had previously demonstrated the feasibility of elevated temperature staging for autoclave prepregs; specifically Cytec 5276-1 [7]. Our expectation was that, given that EP 2750 was already designed for compression moulding, the processing window of EP 2750 would be more constrained than that of 5276-1. This hypothesis seems to have been supported. The shear strain across the tested viscosity range was smaller for EP 2750 than that for 5276-1 (Figure 7), indicating less flow under similar processing conditions. Further, the plateau of shear strains that Smith [7] reported seems to be significantly smaller for EP 2750, requiring greater precision when staging samples of the compression moulding material. Of note, the highest viscosity samples of EP 2750 we found were those considered to be gelled and therefore are not a valid comparison (Figure 7).

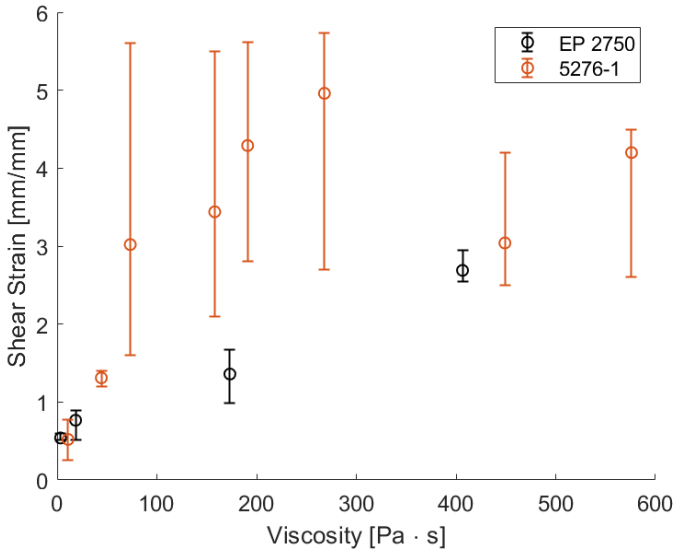

Figure 3. Comparison of shear flow between EP 2750 and 5276-1 [2]

5. Conclusions

Through this flow compaction experiment, we demonstrated that EP 2750 is recyclable using elevated temperature staging. We found a shear flow window when properly staged that should be suitable for compression moulding applications, and should not significantly increase its degree of cure once staged. That said, more work should be done to better characterize the processing window, particularly as it relates to better viscosity modelling and flow compaction under different compaction rates and processing temperatures.

6. Acknowledgements

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7. References

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