PROPERTIES OF THERMOFORMED LOW DENSITY GLASS REINFORCED THERMOPLASTIC SHEET

P.J. Bates, Royal Military College of Canada
A. Chesney, V. Bertrand, Queen's University
M-A Rainville, National Research Council of Canada
E. Haque, AZDEL Inc.

Abstract

The thermoformability of low-density polypropylene sheets reinforced with long discontinuous glass fibers was assessed using a laboratory scale thermoforming machine. The effects of material parameters (glass fiber loading and sheet basis weight), and processing parameters (sheet temperature, and pressure) on part thickness, glass fiber distribution and mechanical properties were evaluated. The results indicate that, for the parts studied, pressure assist is required for thermoforming. Part characteristics were observed to be reproducible and constant over a wide range of sheet temperatures and pressure assist levels. The mechanical properties of the thermoformed sheets were assessed using flexural testing. The modulus and strength values observed were comparable to properties obtained on compression molded samples of the same thickness.

Introduction

Glass mat thermoplastic sheets (GMT) consist of continuous or long discontinuous reinforcing fibers dispersed in a thermoplastic matrix. The thermoplastic matrix of choice is polypropylene due to its low cost per unit volume and adequate thermal properties [1]. A number of techniques are used to manufacture GMT [2, 3]. They include melt impregnation and slurry deposition. A low density GMT has recently been developed using a proprietary manufacturing process that allows the incorporation of air into the composite material [3]. Applications include car headliners made using matched mold processing. This study examines thermoforming of low density GMT sheet in order to determine if this is a viable processing method for this material.

Material and Characterization

The sheets used in this study were manufactured by AZDEL using a proprietary foaming process that combines chopped glass fibers and dispersed polypropylene powder into sheets [3]. The sheets produced are very porous and have a very low basis weight (600-2000 g/m^2). A scanning electron microscope picture is shown in Figure 1 and clearly shows the sheet's porosity.

The three types of sheet used were: 1345 g/m^2 basis weight at 55 wt % glass loading, 945 g/m^2 at 55% glass loading, and 945 g/m^2 at 42% glass loading.

One face of each sheet was covered by a thin non-porous film that served as a binder between the headliner carpet and the low-density composite material. It should be noted that no carpet was used in this work. The opposite face was covered by a non-woven porous scrim to minimize glass fibers on the surface. The scanning electron microscope picture in Figure 1 shows the film on the left and the scrim on the right.

In order to determine the processing temperature range necessary for the forming process, differential scanning calorimetry tests were performed using a Perkin-Elmer calorimeter. These tests were carried out from -100^{o}C to 240^{o}C. All types of sheet were tested, with and without film. The melting temperature of the sheet was observed to be 161^{o}C. The film displayed a melting exotherm above 200^{o}C. Thermoforming temperatures were thus chosen to be 180 and 190°C. These temperatures allow the polymer composite mat to melt and deform while the film remains intact to act as an impermeable air seal during thermoforming.

Thermoforming

Experimental Procedure

Sheets were prepared by cutting the material into 22.9 cm x 22.9 cm (9 in x 9 in) samples. Each sheet was marked with an arrow to indicate the machine direction. This arrow was used to index the samples in the thermoformer.

All samples prepared for the thermoforming trials were gridded by hand, using a 1.25cm (½ in) space between each line. The origin was defined at the center of the sheet, with the *y*-axis being in the machine direction, and the *x*-axis in the transverse direction.

Samples presented in this study were formed using an aluminum mold containing a pie shaped cavity. The cavity was nominally 12.7 cm (5 inches) in diameter on the top and 9.5 cm (3.75 inches) in diameter on the bottom and had a depth of 3.2 cm (1.25 inches).

Thermoforming these parts involved applying a vacuum from inside the cavity and, if necessary, a pressure assist from a pressure box located above the sample. The sample itself was clamped in an aluminum frame situated between the pressure box and mold.

Four net pressures (difference between pressure in the cavity and pressure box) were chosen for the trials: 1 bar (pure vacuum), 2 bar, 3 bar, and 4 bar. Thermoforming trials were carried out for each net pressure, temperature and material combination. Three 3 repetitions were performed to verify reproducibility. Table 1 shows the material and processing parameters.

The oven used for heating the sheets was set to a constant 300^{o}C. There were six heating zones above the sheet and six below. Each zone contained three ceramic infra-red heating elements of 650 watts. The elements were located approximately 14 cm from the sample. The time that each sheet was left in the oven varied depending on its glass loading, basis weight, and desired target temperature. The time was determined with the aid of thermal images.

The pressure thermoforming sequence started with preheating the sheets in a 300^{o}C oven for 1 to 2 minutes. The mold and pressure box then closed on the sheet. A vacuum from the bottom and a pressure from the top were applied for 30 seconds. Air pressure from the bottom was then applied for 0.5 seconds to eject the deformed sample from the mold. When the mold and pressure box were removed from the sheet, a cooling fan blew ambient air on the final part for 20 seconds. This whole process is automated including the activation of the vacuum.

ASTM D790 flexural testing was performed on samples cut from the bottom of the thermoformed sample in both machine and transverse directions. The samples were of nominal dimensions 51 mm x 13 mm x thickness.

Results – Early learnings

Initial thermoforming trials were performed without the impermeable film. The composite was, however, too porous to maintain a net pressure force on the sample and no deformation occurred. All subsequent trials were performed with the impermeable film in place.

Forming trials were then attempted in the shallow cavity using 1 bar net pressure (pure vacuum) at 190°C. Only one of the three sheets, the 945 g/m^2 at 42% loading, would conform to the shallow cavity under these conditions. All other sheets froze before reaching to the cavity surface. However, net pressures of 2 bar and above, obtained using pressure assist, were sufficient to deform all materials. Research reported in subsequent sections was then carried out with pressure assist in addition to the vacuum.

Results – Thickness Profiles

The initial and final thicknesses along the *x* and *y* axes were measured every 1.25 cm in using a Magna-Mike 8500 Hall Effect Thickness Gage. The normalized thickness was then determined by dividing the final thickness by the initial thickness. The effects of temperature, net pressure, basis weight, and glass loading on the thickness profiles are shown on Figures 2, 3, 4, and 5. Each curve shows the normalized thickness profile across the machine (y-axis) direction. The sample thins out to approximately 20% of its initial thickness in the center regardless of material or operating parameter.

The cause of this near constant center thickness may be related to packing of the random glass fibers. The net thermoforming pressure forces air out of the GMT sample until fibers come into contact with one another to balance the compressive pressure force. To better understand this thinning phenomenon, compression tests were performed on a Carver® 12” hydraulic press. These tests compressed 75 mm x 75 mm squares of the three materials between flat plates at a temperature of 190°C. The thickness of the sheets was recorded as a function of time by a Labview® data acquisition system. Typical results for a pressure of 3 bars are shown in Figure 6. It shows that the normalized thickness decreased rapidly from a value of 1 to near constant values in the range 0.25 to 0.28 for all three materials used in this study. These near constant values correspond to thicknesses at which the applied hydraulic pressure is balanced by contact between the glass fibers in the mat. These values are slightly higher than the 0.2 normalized thickness observed experimentally on the thermoformed sheets for each material. This would imply that some thickness reduction occurs in a low density composite sheet due to the increased area the thermoformed material must occupy and further thickness reduction occurs by compression of the porous material.

Results – Glass Fiber Profile

Each 1.25cm x 1.25 cm square along the right side of the machine direction (*y* axis) was cut out. For each square, a pyrolysis test was performed in a 500-600^{o}C muffle oven to determine its glass fiber level after thermoforming. Typical results for the 945 g/m^2 – 55 wt% glass mat are shown in Figure 7. The results show no statistically significant change in glass fiber content compared with unformed material. This suggests that the polymer did not percolate upon pressure thermoforming. Resin percolation refers to the flow of resin through or along the fibers in the composite [4]. It should be noted that the measured glass content of this mat was 47% not 55% because of the diluting effect of the polymer film and scrim.

Mechanical Properties

The flexural strength and flexural modulus of samples cut from the bottom of the thermoformed parts can be compared with literature values obtained on compression molded samples of the same thickness [5]. Figures 8 and 9 show the transverse flexural strength and modulus of thermoformed samples of 1345 g/m^2/55% GF material formed at a net pressure of 2 bars and a temperature of 190°C as a function of thickness. Literature values are shown for reference. The results suggest that thickness has the largest effect on flexural properties and that the type of process used to obtain the thickness is of secondary importance.

It is therefore not surprising that thermoforming pressure and temperature had little effect on the final flexural properties. Typical results for the flexural strength and modulus of the 1345 g/m^2/55% GF material are shown in Figures 10 and 11. Of note is the slight anisotropy between the machine and transverse direction.

The difference in flexural properties among the three thermoformed materials is shown in Figures 12 and 13. No significant difference in yield strength is observed but, as expected, the 42% GF filled material has a lower modulus.

Although there is little difference in yield strength and modulus between the two 55% glass reinforced materials once thermoformed, non-normalized properties of the thermoformed part are affected by basis weight due to differences in absolute thickness. In a purely flexural deformation:

$$\sigma = \frac{3}{2}\frac{P\,L}{b\,d^2} \qquad [1]$$

Where σ = stress, P = load, L = span, b = width and d = thickness. The load required to cause the material to break is thus proportional to:

$$P \propto \sigma \; d^2 \qquad [2]$$

When the product of yield stress and final thickness squared is calculated for each material, the advantage of the higher basis weight material is clearly observed in Figure 14.

Conclusions

Thermoforming experiments were carried out with three different types of low density glass reinforced polypropylene sheet materials. The results show that this composite can be thermoformed. The normalized thickness profiles obtained show little effect of material parameters (glass content or basis weight) or processing parameters (pressure or temperature) suggesting a wide processing window.

There are however several important aspects that potential thermoformers of this material must take into account:

- The low density glass reinforced composite is porous and requires an impermeable membrane to maintain a net pressure force on the sheet. In this research, a thin film of melting temperature approximately 20-40C° above processing temperature worked adequately.

- The stiffness provided by the glass fibers may require more than a simple vacuum for forming to occur. Given the surface area and thickness of the sheet as well as the depth of the cavity, a 1 bar pressure assist in addition to a full vacuum was required to form all composite materials.

- The minimum thickness of the thermoformed material was of the order of 20% of the initial thickness for all material and process conditions tested. This is due to a combination of thickness reduction during forming and air being forced from the mat under pressure until fiber-fiber contact prevents further compression. The part must be designed such that this thin final thickness still meets functionality requirements.

- As a first approximation, the flexural properties of the thermoformed part can be estimated from the final thickness of the component and mechanical property data on compression molded samples of comparable thickness.

Acknowledgements

The financial support of the Natural Science and Engineering Research Council and the Academic Research Program of the Department of National Defense are also gratefully acknowledged.

References

1. Handbook of Polypropylene and Polypropylene Composites", Editor Karian H. G., Marcel Dekker, Inc., New York, (1999).

2. Karger-Kocsis, J. “Swirl Mat- and Long Discontinuous Fiber Mat- Reinforced Polypropylene Composites – Status and Future Trends”, International Symposium on Polymer Composites Science and Technology, 77-92 (1999).

3. Haque, E. and Ickes S., “Development of Low Density Glass Mat Thermoplastic Composites for Headliner Applications”, SAE 2000 World Congress, Detroit, (2000).

4. Flow and Rheology in Polymer Composites Manufacturing, Chapter 13: Sheet Forming of Composite Materials,. Editior Ó.Brádaigh C.M, Elsevier Science, (1994).

5. http://www.azdel.com/superlitedatasheets/SL551200-101.pdf

Keywords

thermoforming, GMT, low density, AZDEL

Table 1 – Material and processing parameters

Material parameters		Processing parameters	
Basis weight (g/m^2)	Glass content (%)	Sheet Temperature (^{o}C)	Net Pressure, (bar)
945, 1345	42, 55	180, 190	1, 2, 3, 4

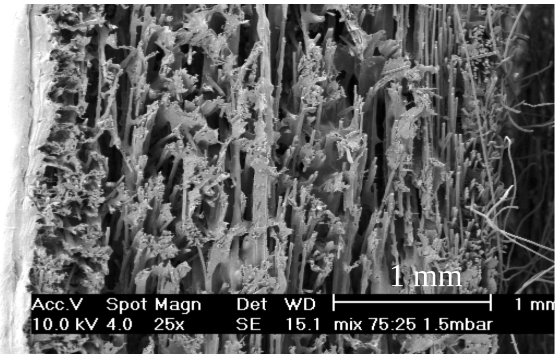


Figure 1 - A cross-sectional view of an unformed low density GMT

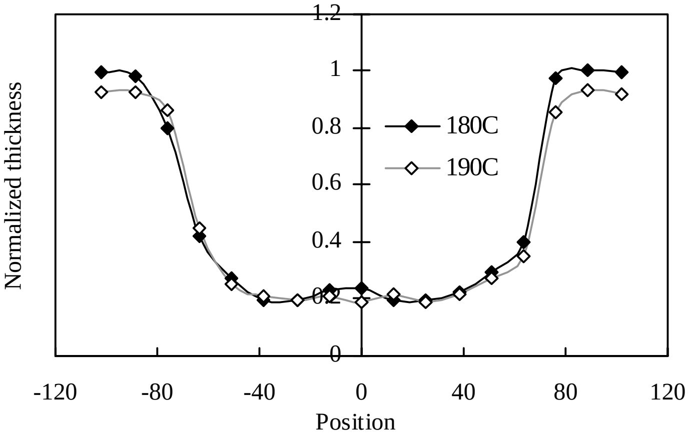


Figure 2 - Normalized thickness versus position as a function of temperature for a 945 g/m^2 basis weight and a 55% reinforcement material processed at a net pressure of 3 bars.

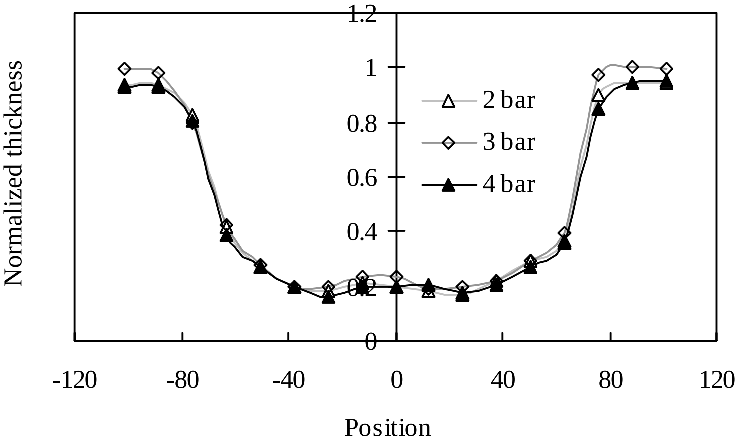
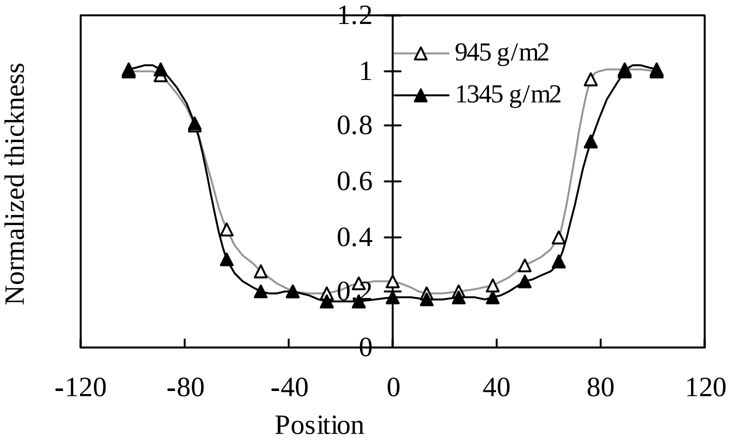


Figure 3 - Normalized thickness versus position as a function of pressure for a 945 g/m^2 basis weight and a 55% reinforcement material processed at a temperature of 180°C

Figure 4 - Normalized thickness versus position as a function of basis weight for a pressure of 3 bars, a temperature of 180°C and a 55% glass fiber reinforced material.



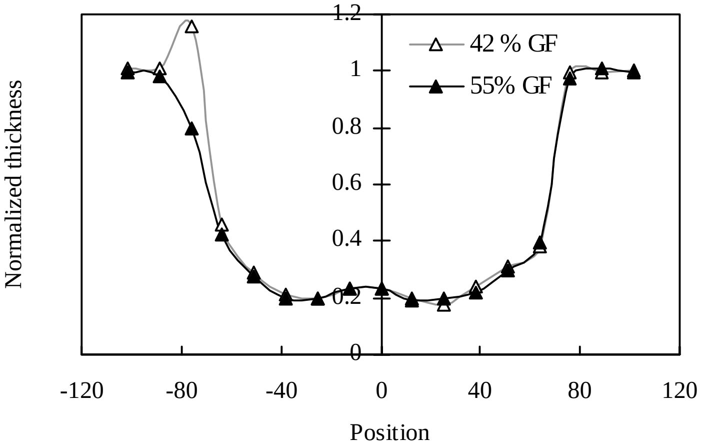
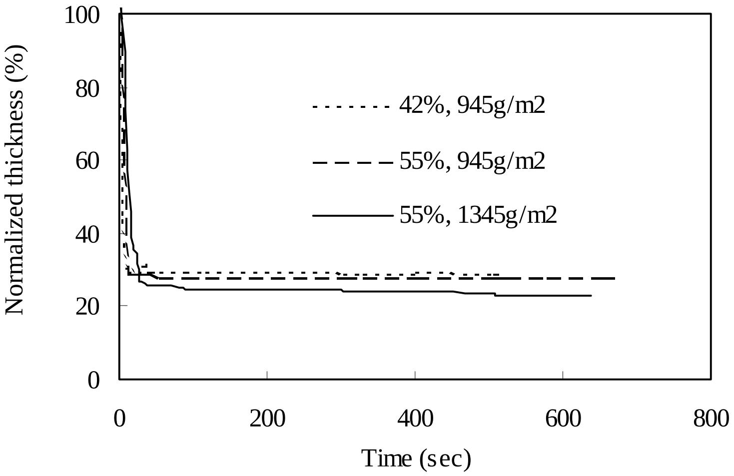


Figure 5 - Normalized thickness versus position as a function of glass fiber content for a basis weight of 945 g/m^2, a pressure of 3 bars and a temperature of 180°C.

Figure 6 – Normalized thickness versus time for compression molded sheets of low density composite material at a pressure of 3 bars.



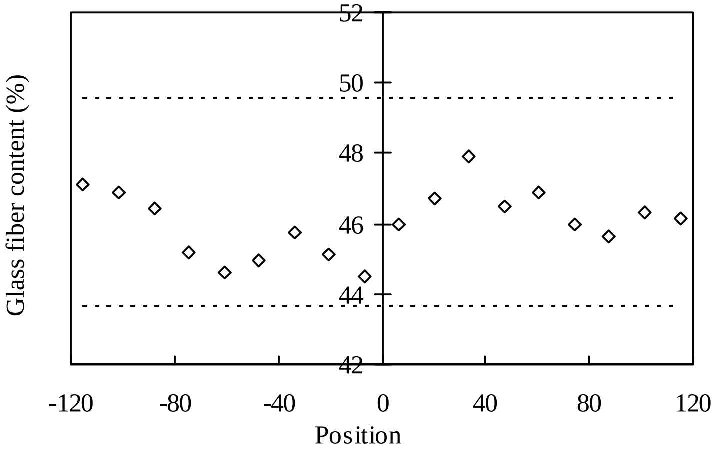


Figure 7 - Glass fiber content versus position for a material of basis weight of 945 g/m^2, processed at a pressure of 3 bars and a temperature of 180°C. The dashed lines represent the upper and lower limits of the glass fiber content measured in the bulk material prior to processing.

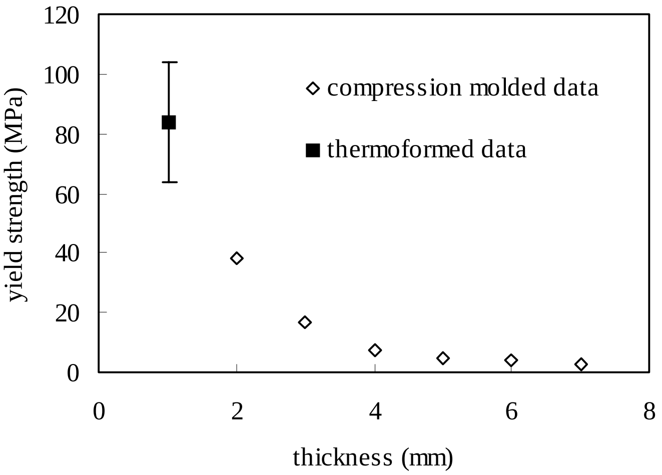
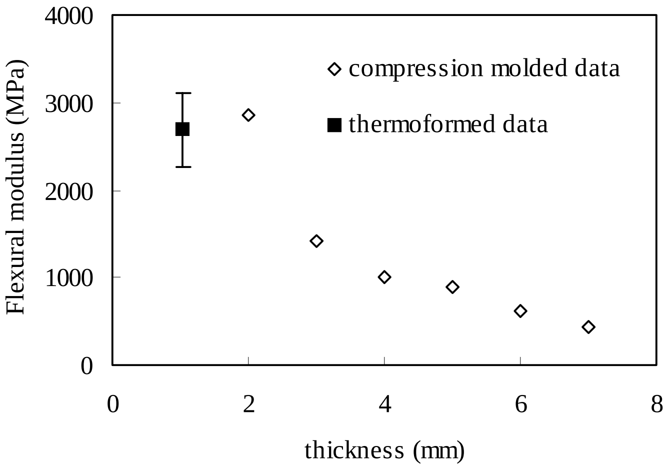


Figure 8 – Transverse flexural yield strength of low density thermoplastic composite (basis weight 1345 g/m^2 and 55% glass fiber) thermoformed at a pressure of 2 bars and a temperature of 190°C and of the same compression molded material data as a function of thickness.

Figure 9 – Transverse flexural modulus of low density thermoplastic composite (basis weight 1345 g/m^2 and 55% glass fiber) thermoformed at a pressure of 2 bars and a temperature of 190°C and of the same compression molded material data as a function of thickness.



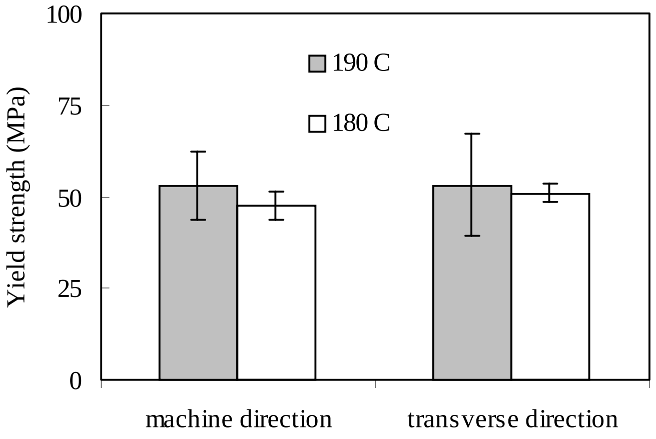


Figure 10 – Yield strength of low density thermoplastic composite (basis weight 1345 g/m^2 and 55% glass fiber) thermoformed at a pressure of 3 bars and at two different temperatures as a function of test direction

Figure 11 – Yield strength of low density thermoplastic composite (basis weight 1345 g/m^2 and 55% glass fiber) thermoformed at a temperature of 190°C as a function of pressure and of test direction

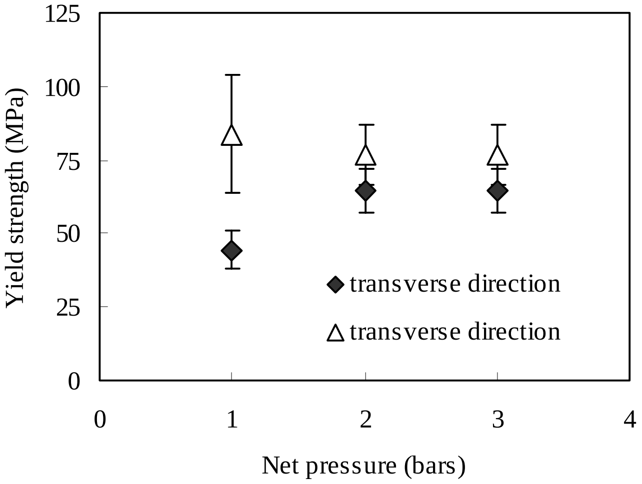
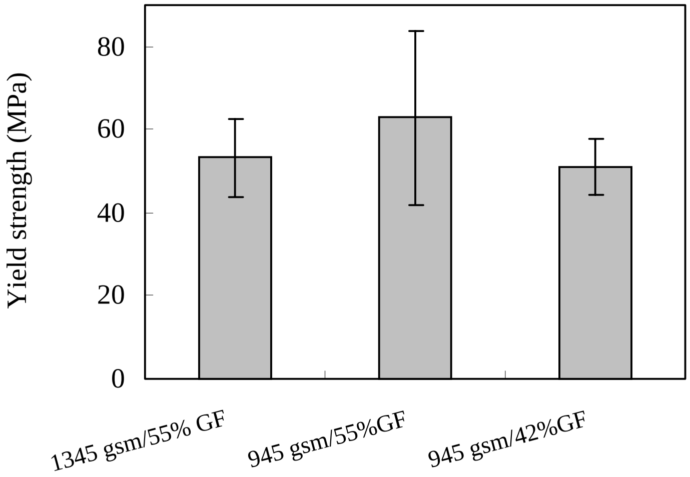


Figure 12 – Yield strength in the machine direction for 3 different types of low density composite. All materials were thermoformed at a temperature of 190°C and a net pressure of 3 bars



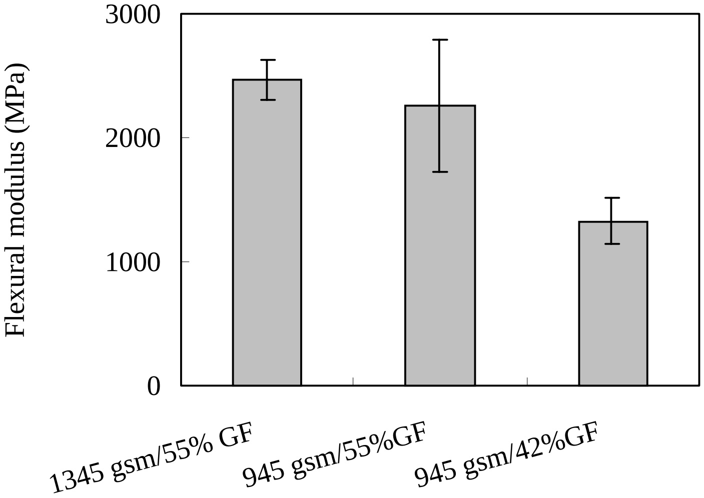


Figure 13 – Flexural modulus in the machine direction for 3 different types of low density composite. All materials were thermoformed at a temperature of 190°C and a net pressure of 3 bars

Figure 14 – Yield stress (in machine direction) x thickness squared for 3 different types of low density composite. All materials were thermoformed at a temperature of 190°C and a net pressure of 3 bars