

EVALUATION OF THE IMPREGNATION CHARACTERISTICS OF CARBON FIBER-REINFORCED COMPOSITES USING POLYPROPYLENE GEL-FILM

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Abstract

Carbon fiber reinforced polypropylene (CFRPP) has been widely used in many engineering fields because of its high specific strength, stiffness, recyclable and lightness. However, polypropylene (PP) is difficult to impregnate into fiber bundles because of its high molten viscosity. In this study, we fabricated CFRPP by using a high speed resin transfer molding (RTM) with PP gel-film whose flow direction is through-thickness for the preform to solve the low impregnating property of the CFRPP. PP gel-film was fabricated by using the quenching method to obtain an amorphous PP which leads to low melting temperature. The crystallinity and thermal property of PP gel-film were analyzed by X-ray Diffraction (XRD) and Differential scanning calorimetry (DSC), respectively. The viscosity of the dissolving state PP was measured by viscometer. The effect of the high speed RTM with PP gel-film on interlaminar shear strength of the CFRPP was compared with a conventional film stacking method by performing the short beam shear test.

1. Introduction

In recent years, carbon fiber reinforced thermoplastic composites (CFRTP) have been widely studied for the application and development of eco-friendly materials in line with the rising environmental awareness worldwide [1-3]. The carbon fiber reinforced thermoplastic composites have several advantages such as environmental superiority, low cost, and high specific strength, compared to carbon fiber reinforced thermoset composites. However, the use of most CFRTPs in fabrics suffers a significant difficulty about their fabrication. This means that the molten viscosity of thermoplastic resin is extremely high compared to that of thermoset resin, which makes it difficult to impregnate thermoplastic resin into fiber bundles [4]. Therefore, a key issue is the achievement of good impregnation of the fabric with the thermoplastic polymers [5].

Generally, various methods have been used to manufacture CFRTPs, such as film stacking, powder impregnation and solution impregnation. Film stacking is one of common methods to make CFRTPs [6-8]. Powder impregnation was developed to promote fiber impregnation and this method is appropriate when the diameter of polymer particles is similar to that of the fiber. But this is difficult to achieve, as the particle size is frequently limited by economic considerations and the effectiveness of the method used to prepare the powder [9]. Solution impregnation requires solubilization of matrix polymer at a suitable concentration followed by the immersion of the fiber within the solution. Wu et al. investigated a solution processing technique and the mechanical properties of carbon fiber reinforced polyethersulfone composites [10]. However, this method is difficult to dissolve a large amount of thermoplastic polymer in common solvents at room temperature because of its high solvent resistance

and semi-crystalline structure. Additionally, the solvent can be difficult to completely remove after impregnation, which can cause many voids [11]. Resin transfer molding (RTM) process has been widely used in various industries because products can be manufactured easily and the cost for manufacturing is lower than those of other manufacturing methods [12]. However, this method has been limited to fabricate thermoset composites because it requires low viscosity of matrix polymer.

In this study, a novel manufacturing method using RTM with PP gel-film was proposed to improve the degree of impregnation and the interfacial characteristics between the carbon fabric and the PP matrix. The PP gel was fabricated by the liquid nitrogen quenching method to impregnate the PP in to carbon fiber bundles at a relatively low temperature ($\sim 140^{\circ}\text{C}$) and the melting temperature of PP gel was characterized by differential scanning calorimeter (DSC). The carbon fiber reinforced PP (CFRPP) composites were fabricated using the film stacking and the high speed RTM whose flow direction is through-thickness for the preform. To investigate the effect of the fabricating methods on the material property of the composites, the interlaminar shear strength (ILSS) were measured by performing short beam shear test. The degree of impregnation was evaluated by SEM images with respect to fabricating method.

2. Experimental

2.1. Materials

PAN-based CF fabric (12K plane weave, AKSA, Turkey) sized with bisphenol A diglycidyl ether epoxy by the manufacturer was used as the composite reinforcing material. Isotactic polypropylene (427888, Sigma-Aldrich Co. LLC., USA) with $M_w = 25 \times 10^4$ g/mol, $M_n = 67 \times 10^3$ g/mol and $T_m = 160 \sim 165^{\circ}\text{C}$ was used as the thermoplastic matrix.

2.2. Fabrication of the PP gel-film

The PP gel was fabricated using isotactic PP (i-PP). The i-PP should be dissolved in solvent at high temperature because of its high solvent resistance. Accordingly, the i-PP was dissolved in ortho-dichlorobenzene at 130°C for 1 h to prepare a solution at 20 wt%. The heated PP solution was quenched by the addition of liquid nitrogen to maintain the amorphous state of PP at low temperature. The quenched PP solution was kept in the freezer for 24 h at -16°C to obtain an amorphous PP gel, as shown in Fig. 1. After fabricating the PP gel, analysis with DSC was performed to check the melting temperature of PP gel. The PP gel was used to make a PP gel-film of 0.6 mm thickness applied press for the uniform impregnation.

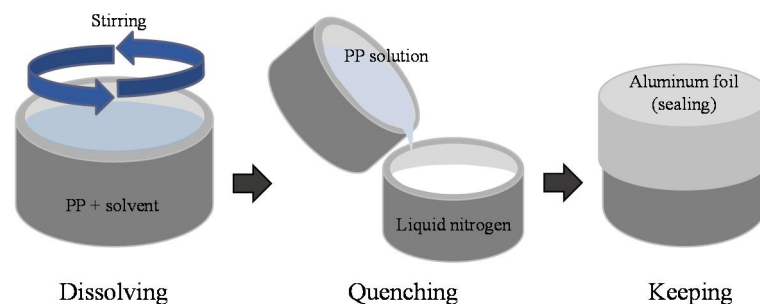


Figure 1. Fabricating process of the PP gel.

2.3. Through-thickness RTM process

The CFRPP composites were fabricated using the RTM method. ten sheets of the PP gel film and CF fabric were stacked alternately and then applied vacuum at 140°C under 1 bar of pressure, as shown in Fig. 2.

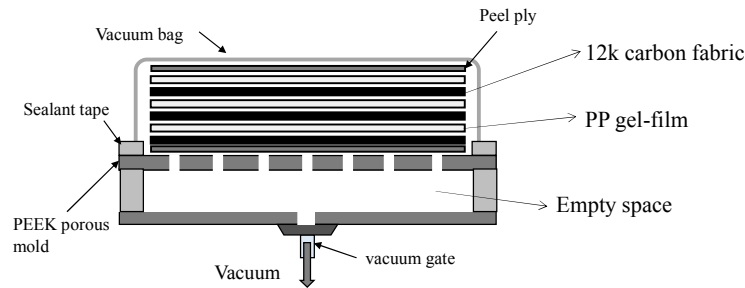


Figure 2. Schematic diagram of the high speed RTM process.

2.4. CF/PP composite fabrication

The CFRPP composites were fabricated by using the film stacking method. The i-PP pellets were used to make an i-PP film 0.2 mm in thickness using a hot press at 220°C and 1 MPa. Eleven sheets of the i-PP film and ten sheets of the carbon fabric with film stacking sequence of $[(0,90)_s]_s$ were stacked alternately and then pressed at 220°C under 20 bar of pressure for 10 min. Fig. 3 shows the molding cycle of the CFRPP composites.

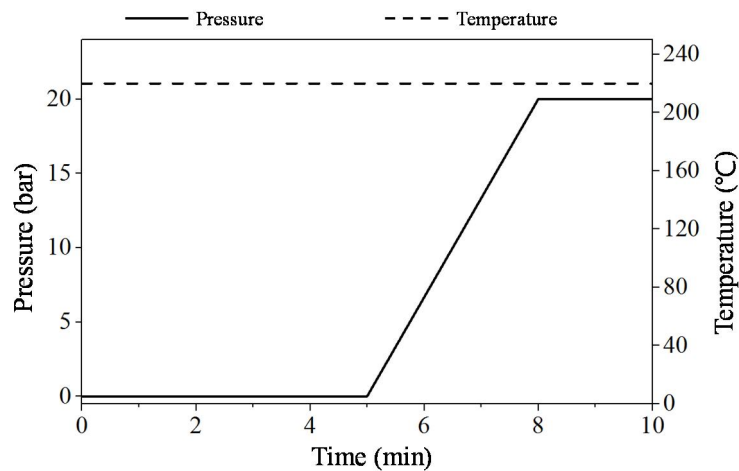


Figure 3. Molding cycle for fabricating the CFRPPs.

2.5. Short beam shear test on the composites

Short beam shear testing is used for the determination of the apparent interlaminar shear strength (ILSS), but it provides information about the quality of adhesion at the fiber/matrix interface. The ILSS of the CFRPP composites were measured using a universal testing machine (INSTRON 5969, MA, USA) based on ASTM D2344. The specimen size was approximately 8 mm × 24 mm × 4 mm and the loading speed was 1 mm/min. the maximum shear strength (τ) was calculated using the following equation:

$$\tau = 0.75 \times \frac{P_m}{bh} \quad (1)$$

Where P_m is the maximum load during the test (N), b is the specimen width (mm), and h is the specimen thickness (mm). The fracture surfaces of the specimens were observed by scanning electron microscopy (SEM) to check the impregnation state of the PP between fibers.

3. Results and discussion

The melting temperature of the PP gel characterized by DSC, as shown in Fig. 4. As can be seen, the PP gel has a characteristic curve at 137°C. This result shows that the melting temperature of PP gel is low compared to PP.

The short beam shear test results of the CFRPP composites are shown in Fig. 5. The ILSS of the RTM specimen increased by 47.3% compared to that of film stacking specimen. This result shows that the impregnation of RTM method was effective than film stacking method.

Fig. 6 shows the SEM images of the composite fracture surface after the short beam shear tests. The film stacking specimen showed poor impregnation state and the PP matrix was completely detached from carbon fiber surfaces due to weak interface as shown in Fig. 6(a). On the other hand, thin PP layers covering the fiber surface were observed from the RTM specimens shown in Fig. 6(b), it seem to be the main evidence for the improvement of the impregnation state when the high speed RTM was applied.

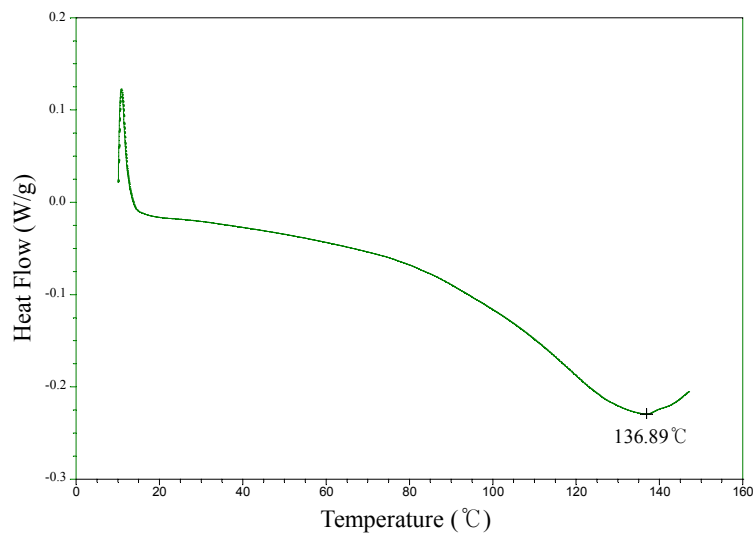


Figure 4. DSC melting curve of the PP gel.

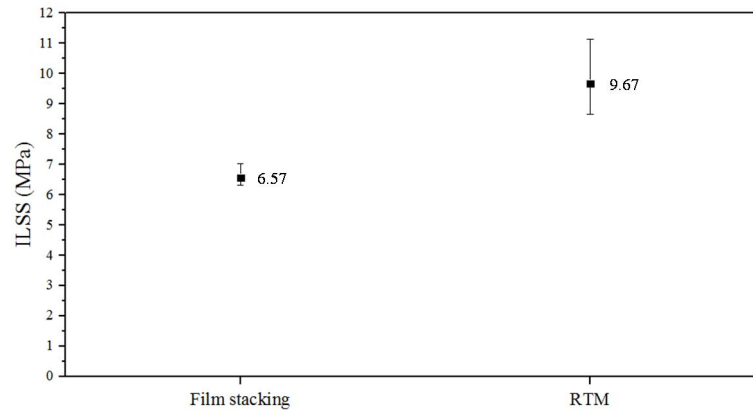


Figure 5. ILSS of CFRPP composites with respect to fabricating method.

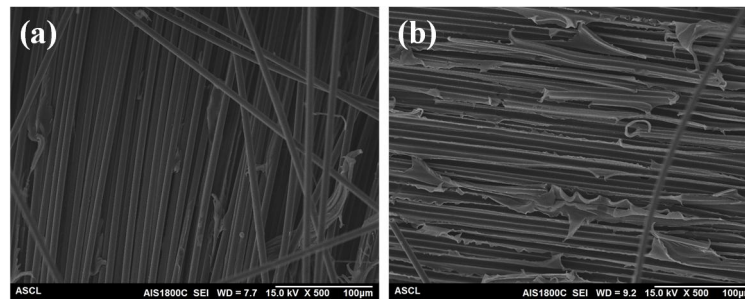


Figure 6. SEM topographies of the composite fracture surfaces: (a) film stacking, (b) RTM.

4. Conclusions

In this study, a new RTM method using a PP gel-film, and the CFRPP composites were evaluated.

The following conclusions were derived from the results:

- The melting temperature of PP gel is lower than PP.
- The ILSS of the fabricated by RTM specimen increased by 47.3% compared to that of the film stacking specimen.
- SEM images showed that impregnation state of the RTM specimen was superior to that of the film stacking specimen.

These results prove that the manufacturing method of RTM that can be used to enhance the interfacial strength between the carbon fiber and PP because of impregnation effect of RTM method.

Acknowledgments

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References

- [1] Wong KH, Mohammed DS, Pickering SJ, Brooks R. Effect of coupling agents on reinforcing potential of recycled carbon fibre for polypropylene composite. *Compos Sci Technol*, 72(7):835–44, 2012.
- [2] Fuad MYA, Ismail Z, Ishak ZAM, Omar AKM. Application of rice husk ash as fillers in polypropylene: Effect of titanate, zirconate and silane coupling agents. *Eur Polym J*, 31(9):885–93, 1995.
- [3] Doufnoune R, Chebira F, Haddaoui N. Effect of titanate coupling agent on the mechanical properties of calcium carbonate filled polypropylene. *Int J Polym Mater*, 52(11–12):967–84, 2003.
- [4] Sakaguchi M, Nakai A, Hamada H, Takeda N. The mechanical properties of unidirectional thermoplastic composites manufactured by a micro-braiding technique. *Compos Sci Technol*, 60(5):717–22, 2000.
- [5] Hou M, Ye L, Lee HJ, Mai YW. Manufacture of a carbon-fabric-reinforced polyetherimide (CF/PEI) composite material. *Compos Sci Technol*, 58(2):181–90, 1998.
- [6] Cattanach JB, Guff G, Cogswell FN. The processing of thermoplastics containing high loadings of long and continuous reinforcing fibers. *J Polym Eng*, 6:345–62, 1986.
- [7] Rijswijk K, Bersee HEN. Reactive processing of textile fiber-reinforced thermoplastic composites – an overview. *Compos Part A – Appl S*, 38(3):666–81, 2007.
- [8] Jespersen ST, Wakeman MD, Michaud V, Cramer D, Manson JA. Film stacking impregnation model for a novel net shape thermoplastic composite preforming process. *Compos Sci Technol*, 68:1822–30, 2008.
- [9] Gibson AG, Manson JA. Impregnation technology for thermoplastic matrix composites. *Compos Manuf*, 4:223–33, 1992.
- [10] Wu GM, Schultz JM. Processing and properties of solution impregnated carbon fiber reinforced polyethersulfone composites. *Polym Compos*, 21(2):223–30, 2000.
- [11] Han SH, Oh HJ, Kim SS. Evaluation of the impregnation characteristics of carbon fiber-reinforced composites using dissolved polypropylene. *Composites Science and Technology*, 91:55–62, 2014.
- [12] Han SH, Cho EJ, Lee HC, Jeong K, Kim SS. Study on high-speed RTM to reduce the impregnation time of carbon/epoxy composites. *Composite Structures*, 119:50–58, 2015.