

MECHANICAL PROPERTIES OF PA6 COMPOSITES REINFORCED WITH SURFACE-TREATED PITCH CARBON FIBERS

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Abstract

Thermoplastic composites with continuous carbon fibers have not been considered as constituent materials for the structural parts as much as the thermosetting composites even though they have good processability, recyclability and low cost. One of the reasons is the poor interfacial adhesion between the reinforcing fiber and the matrix resin. In this work, In order to improve the interfacial adhesion, phenoxy resin-based materials were coated on the surface of the fiber. The interfacial adhesion strengths of the composites were measured to evaluate the effect of the coating materials. The results showed that the composites with the coated pCF had higher interlaminar shear strength than that with neat pCF. In addition, tensile strength which are necessary to secure for the design and analysis of structural composite parts were evaluated.

1. Introduction

Recently, the CFRPs have abruptly attracted much attention again from automotive industries. However, relatively, thermoplastic composites with continuous carbon fibers have not been considered as constituent materials for the structural parts as much as the thermosetting composites even though they have good processability, recyclability [1,2] and low cost. One of the reasons is that the composites don't have enough mechanical properties to be used for structural parts of automotives because of the poor interfacial adhesion between the reinforcing carbon fiber and the thermoplastic matrices [3-8].

In this work, we are interested in fabricating polyamide 6 (PA6) composites with continuous pitch carbon fibers (pCF). Here, in order to improve the interfacial adhesion, phenoxy resin-based materials grafted with pendants having ester functional groups were introduced instead of conventional coating materials for carbon fibers and the effect of the materials on the interface was investigated by evaluating an ILSS of PA6 composites with fabric type reinforcements.

2. Experimentals

2.1. Materials

Polyamide 6 film of a thickness of ~25 μm was purchased from JK Materials (BOPA, South Korea). Two types of phenoxy resins were used as a coating material on the surface of pitch carbon fibers; Phenoxy resin blended with polyester type polyol (PEPO, PKHM-301[®], InChem Co.) and caprolactone-modified phenoxy resin (CPLT, PKCP-80[®], InChem Co.). Plain woven fabric (PF-YSH50A-140, Nippon Graphite Fiber Co.) was used as reinforcement. The strength and modulus of the constituent pitch carbon fiber (XN-50[®], Nippon Graphite Fiber Co.) were 3.9 GPa and 520 GPa, respectively.

2.2. Fabrication of composites

The phenoxy resins were coated on the surface of pCF by dipping a sheet of pCF fabric into a solution followed by drying the fabric at convection oven with 100 $^{\circ}\text{C}$ for 2 hours to completely remove used

solvent. We fabricated composite plates by a process described in the schematic diagram (Fig. 1) using as-received and coated fabrics.

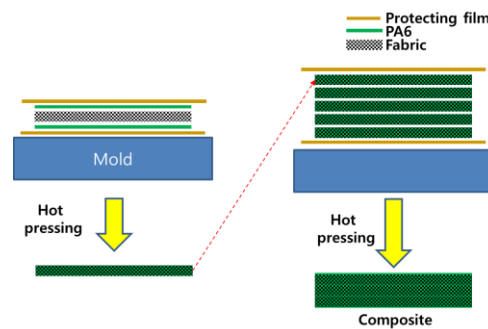


Figure 1. Schematic diagram of the fabrication process.

Before the usage, the PA 6 film was dried one day in a convection oven of 100 °C. At first, each two sheet of PA6 film was placed on both bottom and upper sides of the fabric. Next, after just one minute of aging time at 300 °C, they all were immediately pressed under a pressure for one minute and then cooled down to room temperature. The pressure was around 2.2 MPa. In order to fabricate laminated composites, the prepared intermediate sheets were stacked and pressed for 2 minutes under the same temperature as the aging. The fiber volume fraction (v_f) of composites was calculated at 20.2~21.1%.

2.3 Characterizations and measurement

The amount of the coating resin was measured by thermogravimetric analysis (TGA, Mettler Toledo TGA/DSC1) under N₂ atmosphere from 25 °C to 1000 °C at a rate of 10 °C/min. Interlaminar shear strength (ILSS) and tensile properties of composites was measured according to the ASTM standard 2344 and 3039, respectively.

3. Results and discussion

3.1 Phenoxy-based resins on the fibers

TGA were carried out to measure the amount of a sizing agent on the fiber. From the TGA result, the amount of the agent was estimated at ~ 1.4 wt%. In general, carbon fibers are initially treated by some materials during or after the heat treatment to activate the surface of the fibers. The activation can increase the surface free energy of the fiber which is inherently low. We propose a phenoxy-based coating resin which has a compatible backbone with epoxy groups and a pendant being able to chemically react with free amines of PA. Fig. 2 schematically describes the situation.

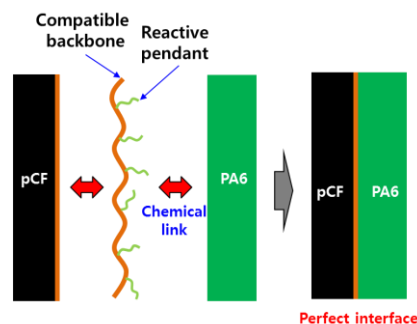


Figure 2. Improvement of the surface between pCF and PA6 with a phenoxy-based resin.

In this work, ester functional groups were used as the reactive pendant and two ester-modified phenoxy resins were coated on the surface of the fibers by dipping a sheet of fabric and drying it. The fractions of PEPO and CPLT in the fabric were 7.5 wt% and 5.2 wt%, respectively subtracting the

amount of the existing sizing agent (1.4wt%) from the raw data of the reduction and considering each percent residue for neat PEPO (6.2%) and CPLT (15.7%). With the fiber density, diameter and the resin fraction, the thickness for each was roughly calculated to 280 nm and 190 nm assuming the uniformity of the resins and the difference of the coating thicknesses was not so high. Therefore, it is thought that the coating is very thin enough to have no effect on the fabrication process and composite properties.

3.2 pCF-reinforced PA6 Composites

Fig. 3 shows the comparison of ILSS for composites reinforced with fabrics coated by different phenoxy resins. As shown in the figure, the ILSS of composites with the coated fabrics were remarkably enhanced by more than 15 %, especially, 20.3 % in case of CPLT-coated fabric compared to the reference composite.

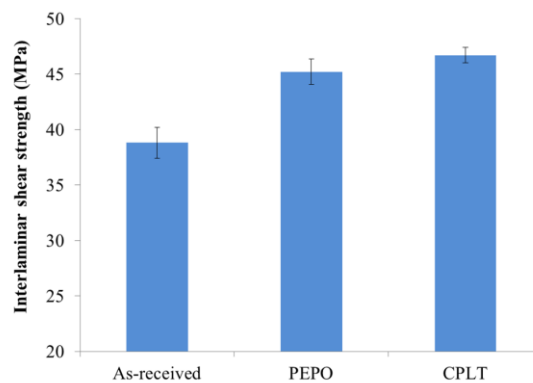


Figure 3. ILSS of composites reinforced with pCF fabrics.

Fig. 5 shows the comparison of tensile strength (TS) and the composite reinforced with CPLT-coated pCF exhibited the highest TS.

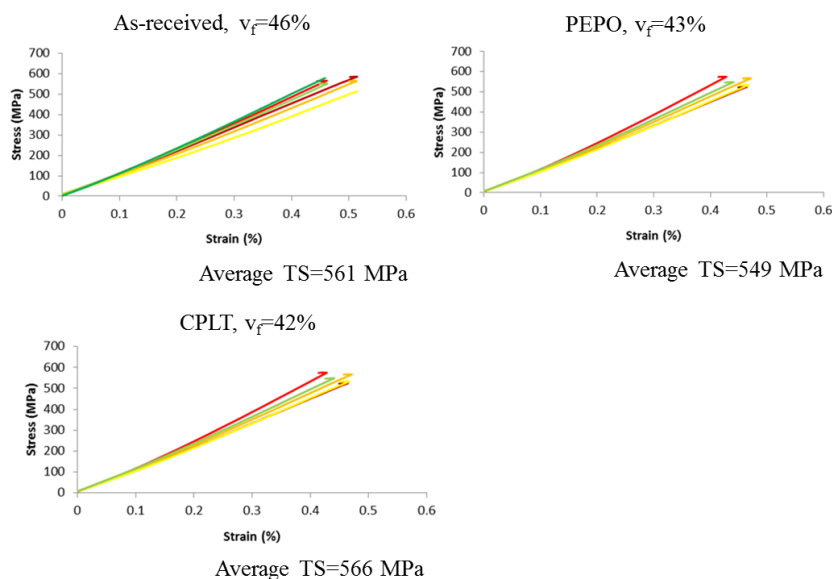


Figure 4. Tensile properties of composites reinforced with pCF fabrics.

4. Summary

In this work, polymer composites were successfully fabricated by a film infusion process with PA6 as

matrix and pCFs as reinforcement. Ester function-modified phenoxy resins were suggested to improve the inherently poor interfacial adhesion. They can be compatible with the sizing of the fiber and simultaneously can form a chemical linkage with the matrix. The results of ILSS show that interfacial adhesion between PA6 and the carbon fiber can be improved by the phenoxy resins and in particular, CPLT effectively enhanced the interfacial adhesion compared to PEPO.

Acknowledgements

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References

- [1] Henshaw JM, Han WJ, Owens AD. An overview of recycling issues for composite materials. *J Thermoplast Compos Mater* 1996 ;1:4–20.
- [2] Ramakrishna S, Tan WK, Teoh SH, Lai MO. Recycling of carbon fiber PEEK composites. Polymer blends and polymer composites. Clausthal Zellerfeld: Trans Tech Publications 1998: 1–8.
- [3] Costa ML, de Almeida SFM, Rezende MC. The influence of porosity on the interlaminar shear strength of carbon/epoxy and carbon/bismaleimide fabric laminates. *Compos Sci Technol* 2001;61:2101-08.
- [4] Fitzer E, Weiss R. Effect of surface treatment and sizing of C-fibres on the mechanical properties of CFR thermosetting and thermoplastic polymers. *Carbon* 1987;25(4):455-67.
- [5] Wang X, Xu D, Liu HY, Zhou H, Mail YW, Yang J, Li E. Effects of thermal residual stress on interfacial properties of polyphenylene sulphide/carbon fibre (PPS/CF) composite by microbond test. *J Mater Sci* 2016;51:334-43.
- [6] Li J, Cai CL. The carbon fiber surface treatment and addition of PA6 on tensile properties of ABS composites. *Curr Appl Phys* 2011;11:50-4.
- [7] Park SJ, Papirer E, Donnet JB. Influence of electrochemical treatment on the surface-properties of carbon-fibers – acid–base character and adsorption enthalpy. *J Chim Phys Phys-Chim Biol* 1994;91:203–22.
- [8] Morgan P. Carbon fibres and their composites. New York: CRC Press, 2005.