

Morphological and mechanical properties of epoxy foam reinforced composites

Kyoko Ishiguro, Takuya Karaki, Samra S. Sangari, and James C. Seferis

University of Washington

Department of Chemical Engineering

Polymeric Composites Laboratory

Seattle, Washington 98195

Abstract

Carbon fiber-reinforced plastics (CFRP) are widely used in a variety of industries such as sports and aerospace. In this work, the effects of presence of foam interlayer in carbon fiber-reinforced laminates on morphological and mechanical properties of the composites were investigated. Carbon fiber-reinforced plastics with epoxy foams were prepared at various processing conditions. The optical microscopy and the scanning electron microscopy (SEM) micrographs showed that the porous composites of pore size of less than 100nm were obtained. The presence of foam in the laminates resulted in composites with a better mechanical strength than the pore-free composites.

Introduction

Polymeric composites materials are used for a wide variety of products that require high strength and low weight [1]. The matrix plays an important role in composites materials. Epoxy foams have gained special attention in composites industry due to their high adherent strengths, low water absorption, good dimensional stability, good heat resistance and generally good resistance to chemical attack [2-9]. The foams in the composites materials can inhibit cracks is worthy of remark.

In this work, we have investigated replacement of the traditional epoxy matrix with epoxy foams to further increase the strength-to-weight ratio. Optical microscopy, field emission microscope and SEM revealed the effect of different processing conditions on foam morphology. Through concurrent mechanical testing, we correlated the effect of foam size and separation on the strength of epoxy-foam composites.

Experimental

Resin Formulation

In this work, EPON[®] 828 from E.V. Roberts and Araldite MY 9655 from Vantico Inc were used as the base epoxy resin for matrix. EPON[®] 828 was based on diglycidyl ether of bisphenol-A (DGEBA). Araldite MY 9655 was a tetraglycidyl methylene dianiline (TGMDA) derivative. The epoxy resins were mixed in a 60:40 ratio

by weight of MY 9655: EPON[®] 828 in the oil bath at 120°C and were stirred at least for 60 minutes until they were completely mixed. For this epoxy ratio, 45 weight ratio Diaminodiphenyl Sulfone (DDS) was weighed and heated to melt. The melt was then added to the epoxy resin as curing agent in the oil bath and the mixture was mixed completely.

Celogen[®] AZ 120 (based on Azodicarbonamide) obtained from Crompton Corporation was incorporated into the epoxy and DDS mixture as foaming agent. Then Pluronic L-64 from BASF Corporation was added as surfactant. In addition, styrene oxide was added as brewing agent from Sigma-Aldrich. The effect of styrene oxide content on the foam size was investigated by adding 1ml, 5ml, 10ml, 15ml and 20ml of styrene oxide to 100g of epoxy mixture. Stir times of 0.5, 1 and 3 hours were set for each sample. All these samples were utilized as the resin for preparation of the prepreg specimens with carbon fibers.

Prepreg

A hot-melt prepreg machine was performed to impregnate carbon fibers with epoxy. Two rolls with pressure of 67kPa and 93kPa were used to facilitate the impregnation process. The prepreg fiber partial weight was set to 150 g/m² and the nominal epoxy content was 30±3 wt% for all experiments. The filming and impregnation temperatures were 82°C and 93°C, respectively. The line speed was at 1.2 m/min and the doctor blade gap was 0.25mm.

Laminates

The laminated samples were placed in an autoclave and cured using various processing conditions in order to obtain CFRPs with different pore size.

For the preparation of the first set of samples the autoclave cure cycle ramped at 0.56°C/min to 23.89°C followed by a 50min hold and then 3.06°C/min ramp to 177°C followed by a 2 hours hold and finally drop in temperature to 24°C at the rate of 2.78°C/min. Pressure was ramped to 310 kPa at 62.0 kPa/min followed by a 3.8 hours hold. For the preparation of the second set of samples, the autoclave cure cycle ramped at the same rate to the same temperature as the first set, but pressure setting

was changed to 110 kPa/min ramp to 552 kPa followed by a 3.8 hours hold.

Surface Analysis

To observe of the sample surface conditions, an AMRAY 1850FE Field Emission Microscope, a JEOL-JSM5200 SEM from JEOL Ltd., and Nikon optical microscopic were used. About 2mm thick of the samples were cut and polished using Poliment® polisher with alumina micro-particles from Buehler Uehler Ltd.

Mechanical Analysis

An Instron 4505 screw testing frame and Instron Series IX software were used to perform 3-point bending test, mode I interlaminar fracture toughness, GIc, and mode II, GIIC, interlaminar fracture toughness. Mode I a double cantilever beam (DCB) method was utilized and mode II the end notch flexure method (ENF) was employed. For each laminate three samples of about 12.0 mm wide were tested; a crack tip was created before testing.

Results and Discussion

The SEM and optical microscopy results indicated that the amounts of foaming agent and surfactant had no significant effect on the epoxy foam size. These results are presented in Table 1.

The Effects of Chemical Amount

The SEM photomicrographs of the samples 1 and 2 exhibited no significant morphological differences although these samples were cured under the same pressure. Both samples had almost the same foam size of about 1 μ m. Some foams of about 30 μ m in diameter were also observed in these samples.

Sample 5 and sample 8 showed no significant differences in their morphology. Bigger foams size of about 250 μ m and above were seen in these samples. The increase in styrene oxide content at a constant pressure resulted in the significant increase of the foam size.

Stirring Time

The SEM and optical microscopy results indicated no significant morphological differences between samples 1 and 12. The foams size of 1 μ m were found in these samples. Stirring for 3 hours resulted in a significant increase of the viscosity of the samples that made the further processing of these samples impossible.

Pressure

Figure 1 indicates that the foam size was controlled by the applied pressure. The macro size foams were observed in Figure 2, while no macro size foams were observed in the samples that were cured under high pressure

setting and 5ml of styrene oxide. From these results, it was suggested that the increase in pressure at a constant styrene oxide content led to a considerable decrease in the foam size. In addition, this assumption was confirmed by the measurement of density of these samples. The increase in the density by increasing the styrene oxide content supported these SEM results. Density of the control sample was 0.0015g/mm³, while the density of sample 9 was 0.0014 g/mm³ and the density of sample 2 was 0.0013g/mm³. This indicated that sample 9 which was cured at higher pressure was heavier than sample 2 that was cured at lower pressure but was lighter than the control sample, meaning that sample 9 contained both micro and nanoscale foams.

Laminate Numbers

It was considered that the number of layers might have affected the foam size due to the pressure created by the weight of the top layers. In addition, the number of layers could also cause an inhibition of the gas escape which in turn influenced the foam size. However the significant effect of laminate numbers was not confirmed by SEM pictures. In contrast the morphological investigations of 8plies and 16plies samples indicated that the number of layers has no significant effect on the foam size.

Mechanical Test Results

The effect of the foam size and ultimately pressure on mechanical properties of control sample, sample 2 and sample 9 were studied. Figures 3 demonstrate the results of 3-point bending test and double cantilever beam test. From the result of the 3-point bending test, no significant difference between control sample and sample 9 were observed, however, when control sample were compared with sample 9 in the weight, sample 9 was lighter than control sample. As shown in Figure 4, no difference between control sample and samples 2 and 9 was observed in GIc. The existence of foam however was expected to affect the mechanical properties of the laminates. The reason why there was no difference in GIc was that cracks proceeded to foams meaning cracks propagation was inhibited by presence of foams and also fiber bridges in the sample and also existence of fiber bridges in the samples.

Figure 5 demonstrated the GIIC of the control sample, sample 2 and sample 9. It was observed that the foam samples had the same fracture toughness characteristics. Moreover, the fracture toughness of the control sample was higher than samples 2 and 9. The foam had a deteriorating effect on the fracture toughness of the samples 2 and 9 although the difference of foam morphologies of these samples was not enough to make any conclusion on GIIC results.

Conclusions

CFRP samples with various amounts of styrene oxide were prepared at different processing conditions. The morphological properties of the uncured and cured samples were studied. There were no significant effects on the foam size by varying the stirring time, foaming agent content and surfactant amount. However, the styrene oxide content and the pressure considerably influenced the size of the foams. The decrease in the styrene oxide content and increase of the pressure led to the decrease in the foam size indicating that the macro foam size can be controlled micro and nano foam size. The difference in foam morphology was not sufficient enough to suggest that formation of foam affected the mechanical properties. The presence of foam in the samples decreased the fracture toughness values. Further work on the precise control of the nanofoam size through optimization of the processing conditions is under investigation.

References

- [1] Mallick, P.K., *Fiber Reinforced Composites: Materials, Manufacturing, and Design 2nd ed.*, (1993), New York: Marcel Dekker, Inc.
- [2] Sigh, K.P., Palmese, G.R., *Journal of Applied Polymer Science*, 91: 3107 (2004).
- [3] Stefani, P.M., Tejeira Barchi, A., Sabugal, J and Vazquez A., *Journal of Applied Polymer Science*, 90: 2992 (2003)
- [4] H. Lee and K. Neville, *Handbook of Epoxy-resin*, (1967)
- [5] V. I. Raman and G. R. Palmese, *Material Research Society Symposium Proceeding*, 788: 347 (2004)
- [6] Bledzki, A.K., Gassan, J., and Zhang, W., *Journal of Cellular Plastics*, 35 (1999)
- [7] Stevens, G.C., Perkins, E., and Champion, J.V., *IEE Conference Publication*, (1998), 298 (Int. Conf. Dielectr. Mater., Meas., Appl. 5th, 1998)
- [8] Soles, C.L., Yee, A.F., *Journal of Polymer Science:Part B:*, 38:792 (2000)

Key Words

Composites materials, Carbon fiber reinforced plastics, foams.

Sample no.	Styrene oxide, ml	Celogen AZ, g	Pluronic L64, g	Pressure, kPa	Stir time, hour	Foam size
Control	0	0	0	310	0	no foam
1	1.0	10	0.1	310	1.0	B, F
2	5.0	10	0.1	310	1.0	B, F
3	5.0	5.0	0.1	310	1.0	B, F
4	5.0	10	1.0	310	1.0	B, F
5	10	10	0.1	310	1.0	A, B
6	10	5.0	0.1	310	1.0	A, B
7	10	10	1.0	310	1.0	A, B
8	15	10	0.1	310	1.0	A, B
9	5.0	10	0.1	552	1.0	C
10	10	10	0.1	552	1.0	B, C
11	20	10	0.1	310	1.0	A
12	5.0	10	0.1	310	0.5	B, C
13	5.0	10	0.1	310	3.0	not measured

A: over 50 μ m , B: between 10 μ m to 50 μ m, and C: under 10 μ m

Table 1. Effects of chemicals and pressure on foam morphology

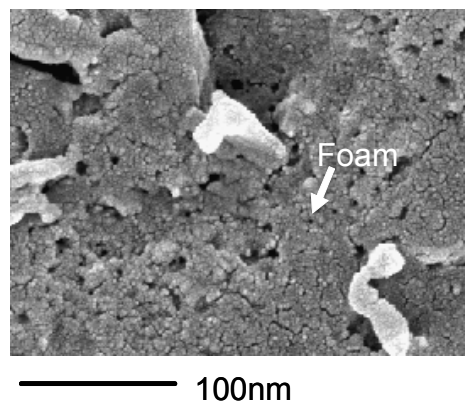


Figure 1: SEM photomicrograph of epoxy foams. (Same resin ratio as sample#9)

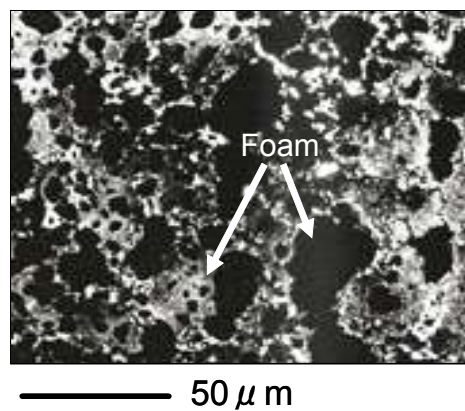


Figure 2: SEM photomicrograph of CFRP with epoxy foam. 50x magnification

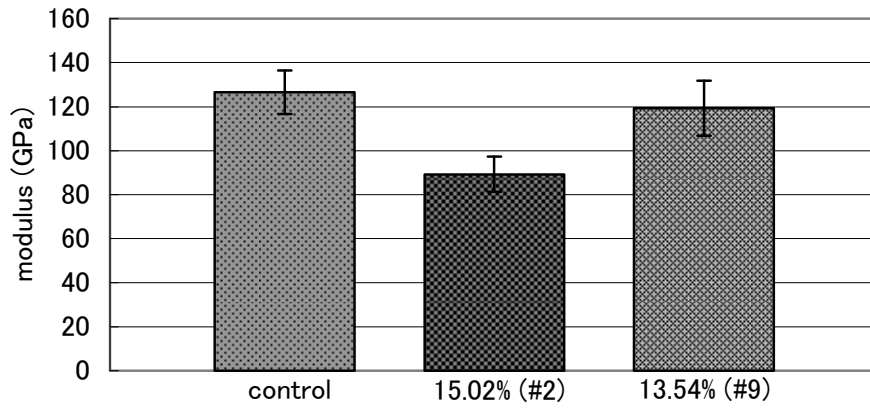
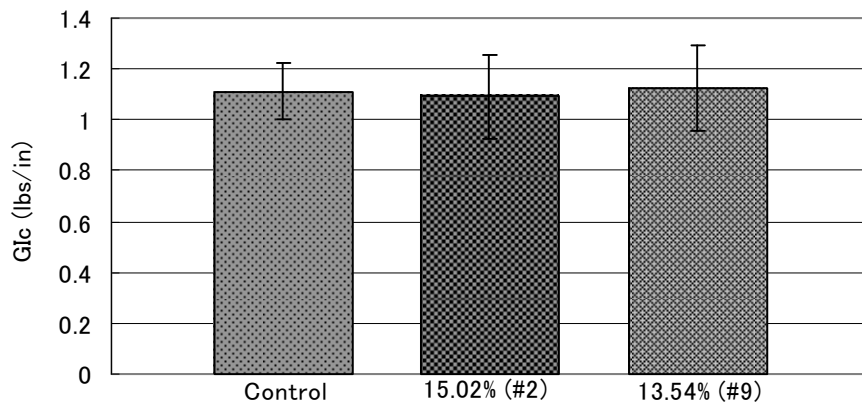
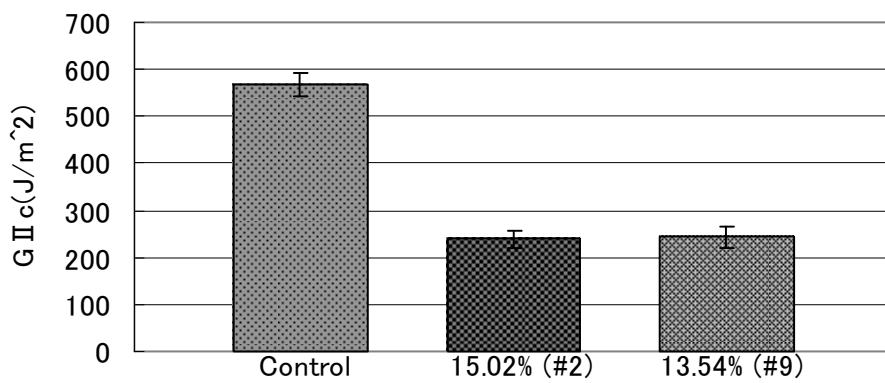


Figure 3. 3-Point Bending Test



**Figure 4. Mode I Fracture Toughness (GIc)
Double Cantilever Beam (DCB)**



**Figure 5. Mode II Fracture Toughness (GIIc)
The End Notch Flexure Method (ENF)**