

THE INFLUENCE ON MECHANICAL PROPERTIES OF EPOXY FOAM MATRIX STRUCTURAL DIFFERENTIATIONS

Kyoko Ishiguro¹⁾, Samra S Sangari¹⁾ and James C. Seferis^{1,2*)}

¹⁾University of Washington
Department of Chemical Engineering
Seattle, Washington 98195

²⁾Polymeric Composites Laboratory
Glocal University
and
Center for Composite Materials
University of Delaware
Newark, Delaware 19716-3144

ABSTRACT

Composite materials are widely used because of their low weight and relatively high strength and stiffness. One type of advanced composite relies on a foam matrix to improve its properties performance. Foam matrices show high adherent strength, low water absorption, good dimensional stability, as well as heat and chemical stability. In this study, nano foam composites were prepared using different processing conditions. The mechanical and morphological properties of the nano foams were studied with the Instron 4505 screw testing frame, the Instron Series IX software and scanning electron microscopy (SEM), respectively. The results showed that nano scale foams were formed in the matrix and high cure pressure improved the mechanical strength of the composites.

KEY WORDS: Cure/ Process- Control Technology, Carbon Fiber Composites, Nano Scale Foams

* Indicates author to whom correspondence should be sent

1. INTRODUCTION

Polymeric composite materials are used for a wide variety of products that require high strength and low weight [1]. The matrix provides many essential functions such as keeping the reinforcing fibers in proper orientation and position so that they can carry load and provide all of the interlaminar shear strength of composites. Epoxy foams have gained special attention in composites industry due to their high adherent strength, low water absorption, good dimensional stability, good heat resistance and generally good resistance to chemical attack [2-8]. A particular advantage of epoxy foams is their tendency to inhibit crack propagation in composite materials.

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

2. EXPERIMENTAL

2.1 Resin Formulation Toray T300YC carbon fibers were used. A mixture of EPON[®] 828 from E.V. Roberts and Araldite MY 9655 from Vantico Inc. were utilized as the base epoxy resin. EPON[®] 828 is based on diglycidyl ether of bisphenol-A (DGEBA) and Araldite MY 9655 is a tetraglycidyl methylene dianiline (TGMDA) derivative.

The epoxy resins were mixed in a 60:40 weight ratio of MY 9655: EPON[®] 828 and stirred until they were completely mixed. 45 weight ratio Diaminodiphenyl Sulfone (DDS) was melted and added to the epoxy system as the curing agent. 10 weight ratios Celogen[®] AZ 120 (Azodicarbonamide) from Crompton Corporation was incorporated into the epoxy and DDS mixture as a foaming agent. Moreover, styrene oxide from Sigma-Aldrich was added as blowing agent. 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 the base epoxy. Then Pluronic L-64 from BASF Corporation that was a block copolymer surfactant was added.

2.2 Stirring An electric stirrer mixer and a high shear mixer were used for studying the relationship between mixing speed and foam morphology. In the case of mixing utilizing the electric stirrer, an oil bath at 120°C was used to reduce the resin's viscosity for easier stirring. Stir times of 0.5, 1 and 3 hours were set for electric stirrer mixing. Also, 10%, 20% and 30% mixing speed were chosen for the high shear mixing to evaluate the dependence of the mixing speed on the foam size and morphology. In this case, the stir time was 1 hour. All these samples were utilized as the resin for preparation of the prepreg specimens with carbon fibers. These conditions are also showed in Table 1.

Table 1. Matrix Mixing Condition

Matrix	Mixing type	Oil bath
(1) Epoxy mixture and additions	Electric Stirrer	120°C
(2) Epoxy mixture and additions	High Shear Mixer (10%)	N/A
(3) Epoxy mixture and additions	High Shear Mixer (20%)	N/A
(4) Epoxy mixture and additions	High Shear Mixer (30%)	N/A

2.3 Prepreg A hot-melt prepreg machine was used to impregnate carbon fibers with epoxy. [9] Two rollers 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.

2.4 Laminates For preparation of the first set of samples, the autoclave cure cycle ramped at 0.56°C/min to 23.89°C followed by a 50 min hold and ramped at 3.06°C/min to 177°C followed by a 2 hour hold, and finally dropped 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 was ramped at the same rate to the same temperature as the first set, but the pressure ramp setting was changed to 110 kPa/min to 552 kPa followed by a 3.8 hour hold.

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

2.6 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 interlaminar fracture toughness (GIIc). [10] For GIc a double cantilever beam (DCB) method was utilized and for GIIc an end notch flexure method (ENF) was employed. [11] For each laminate three samples of approximate 12.0 mm width were tested. A crack tip was created before testing.

3. RESULT AND DISCUSSION

The SEM and optical microscopy results indicate that the amounts of foaming agent and surfactant had no significant effect on the epoxy foam size.

3.1 The Effects of Concentration of Chemicals The SEM photomicrographs showed the effects of curing pressure and styrene oxide concentration on the foam morphology. However, there were no significant effects of the Celogen AZ and Pluronic L64 on the foam morphology. Table 2 shows the effects of styrene oxide concentration and autoclave pressure on foam size. Figure 1 showed that sample 1 had foam diameters

under 10 μ m. It was clear that the resin rich part had a lot of foam. However, the diameters of the foams formed in sample 2 were between 10 μ m and 50 μ m, see Figure 2.

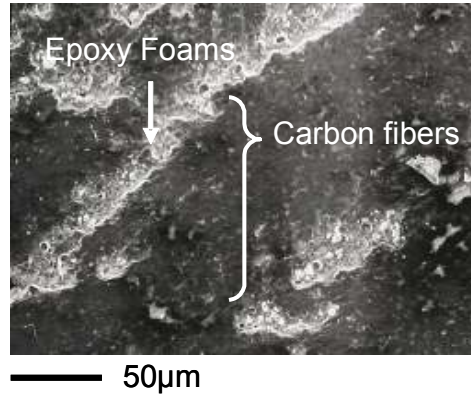


Figure 1: SEM Photomicrograph of CFRP with Epoxy Foam, the electric stirrer was used for the mixing, 350x magnification

Table 2. The Effect of Chemicals and Pressure on Foam Morphology

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	5.0	10	0.1	310	1.0	B, C
2	10	10	0.1	310	1.0	A, B
3	5.0	10	0.1	552	1.0	C

Foam Size A: over 50 μ m , B: between 10 μ m to50 μ m, and C: under 10 μ m

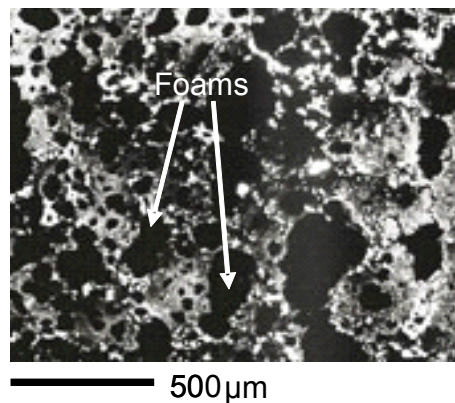


Figure 2: The SEM Photomicrograph of CFRP with Epoxy Foam, the electric stirrer was used for the mixing, 50x magnification

3.2 Stirring In Figure 3, the SEM photomicrographs of sample 3 showed that there were no significant differences between the samples that were made by the electric stirrer mixer and the high shear mixer. Figures 3 a) and 3 b) demonstrate that the epoxy matrix had homogeneous broadening foams. On the other hand, picture 3 c) shows that the foams were placed disproportionate on the left side in this picture that an electric stirrer did not equalize the distribution of foams in the epoxy mixture. Samples that were made by the high shear mixer had diffused foams in the matrix.

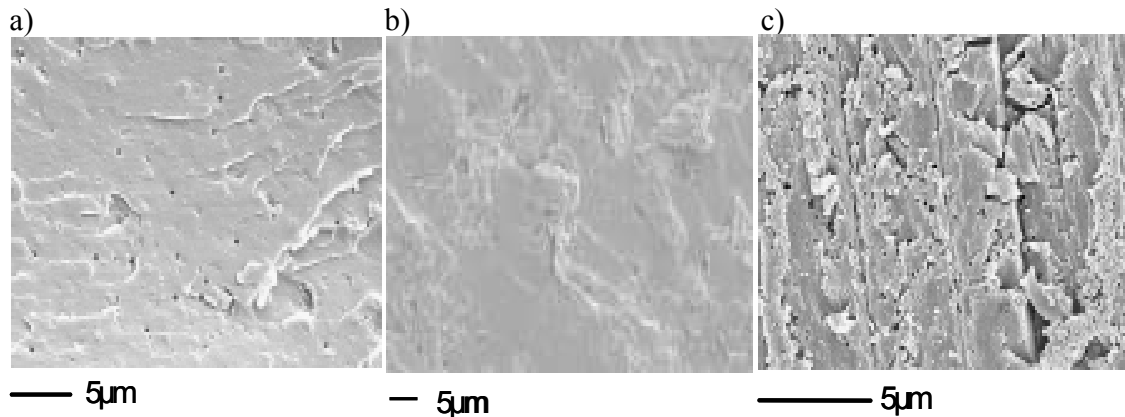


Figure 3: SEM Photomicrographs of Epoxy Foams.

- a) The high shear mixer (10%) 3649x magnification, b) The high shear mixer (30%) 3304x magnification, c) The electric stirrer mixer 5054x magnification.

3.3 Pressure As evident from morphological results foam size was controlled by the applied curing pressure, and 3 showed that. Sample 1 had macro size foams while no such foams were observed in sample 3. These results suggested that increased pressure at a constant styrene oxide content led to a considerable decrease in the foam size. These results were additionally confirmed by density measurements; i.e the increase in styrene oxide content increased the density.

3.4 Void Content Density of the control sample was 0.0015g/mm^3 , while the density of sample 3 was 0.0014 g/mm^3 and the density of sample 1 was 0.0013g/mm^3 . From these numbers, the void content was calculated by the equation below.

$$\text{Void content} = \left\{ \frac{D_c - D_f}{D_c} \right\} \times 100$$

In this equation, D_c is the average density of control samples and D_f is the average density of CFRP foam samples. The CFRP foam samples (sample 3 and sample 1) reduced about 7% and 13% in weight, respectively, compared to the control sample.

3.5 Mechanical Test Results The effect of foam size and pressure on mechanical properties of the control sample, sample 1 and sample 3 were investigated. Figure 4 shows there was no significant difference between the 3-point bending results of control sample and sample 3. However, when control sample is compared with sample 3 by weight, sample 3 was lighter than the control sample.

Figure 5 shows the results of the GIc test. There was no difference in Mode I fracture toughness of the control sample and sample 1. This was due to the fact was that crack propagation was inhibited by the presence of foams. The existence of foam however was expected to affect the mechanical properties of the laminates.

Figure 6 shows the GIIC (Mode II Fracture Toughness) of the control sample, sample 1 and sample 3. The two foam samples had similar fracture toughness characteristics. Moreover, the fracture toughness of the control sample was higher than samples 1 and 3. The foam had a deteriorating effect on the Mode II fracture toughness of the samples 1 and 3, although the difference of foam morphologies of these samples was not enough to make any conclusion on GIIC results.

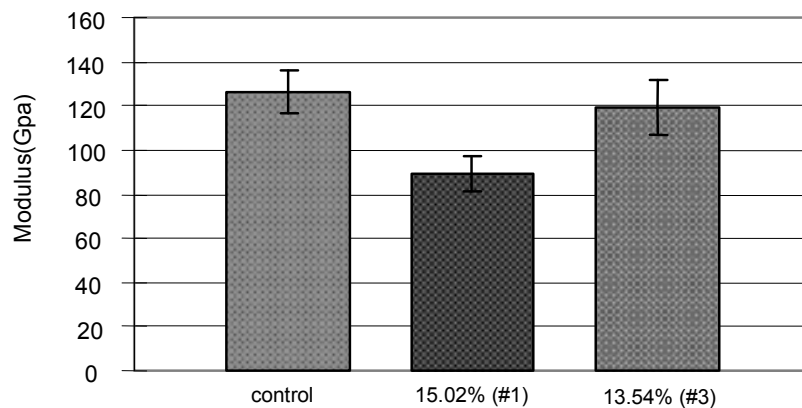


Figure 4. 3-Point Bending

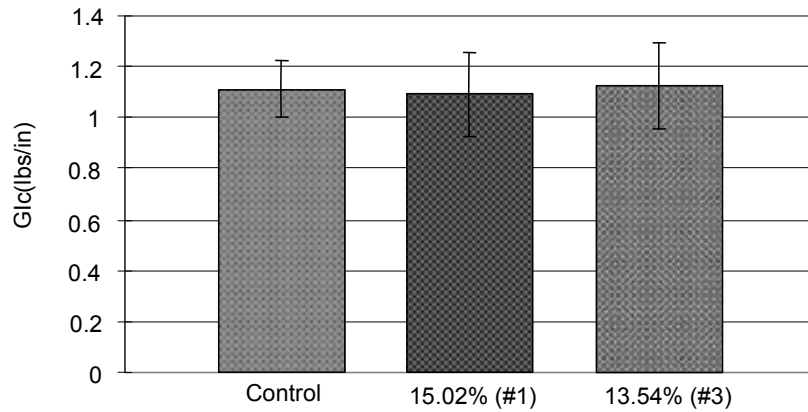


Figure 5. Mode I Fracture Toughness (GIc)
Double Cantilever Beam (DCB)

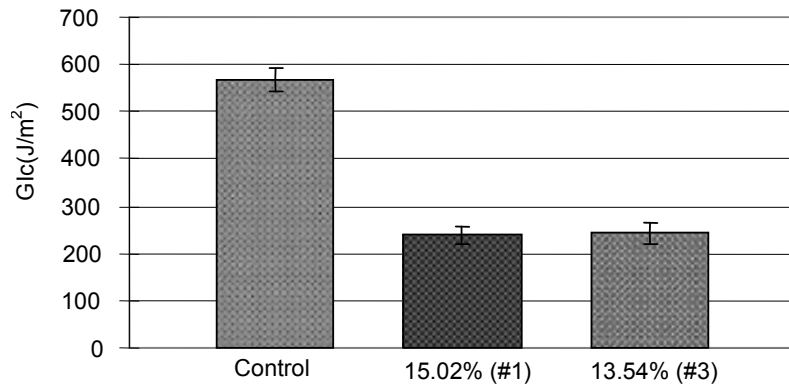


Figure 6. Mode II Fracture Toughness (GIIc)
as measured by The End Notch Flexure Method (ENF)

4. CONCLUSION

In this study, CFRP samples with several amounts of chemicals were prepared at different processing conditions. There were no significant changes on the foam size by varying the stirring time, stirring speed, and foaming agent and surfactant contents. However, the styrene oxide content and the pressure considerably influenced the foam size. A decrease in styrene oxide content and increase of pressure led to a decrease in the foam size indicating that the macro foam size can be controlled down to the micro and nano scale. In addition, a high shear mixer can make more diffuse foams when compared to an electric stirrer. The difference in foam morphology is not sufficient enough to suggest that formation of foam affected the mechanical properties. The presence of foam in the samples decreased the fracture toughness. Further work on controlling nano foam size through process condition optimization is on going.

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