Influence of cure conditions on out-of-autoclave bismaleimide composite laminates

Sudharshan Anandan,[1] Gurjot S. Dhaliwal,[1] V. A. Samaranayake,[1] K. Chandrashekhara,[1] Thomas R. Berkel,[2] Doug Pfitzinger[3]

[1]Missouri University of Science and Technology, Rolla, Missouri, 65409
[2]Boeing Research & Technology, St. Louis, Missouri, 63166
[3]GKN Aerospace, St. Louis, Missouri, 63042
Correspondence to: K. Chandrashekhara (E-mail: chandra@mst.edu)

ABSTRACT: Bismaleimides (BMI) are thermosetting polymers that are widely used in the aerospace industry due to their good physical properties at elevated temperatures and humid environments. BMI-based composites are used as a replacement for conventional epoxy resins at higher service temperatures. Out-of-Autoclave (OOA) processing of BMI composites is similar to that of epoxies but requires higher cure temperatures. Polymer properties such as degree of cure and crosslink density are dependent on the cure cycle used. These properties affect mechanical strength as well as glass transition temperature of the composite. In the current research, carbon fiber/BMI composite laminates were manufactured by OOA processing. The void content was measured using acid digestion techniques. The influence of cure cycle variations on glass transition temperature and mechanical strength was investigated. Properties of manufactured specimens were compared with that of conventional autoclave cured BMI composites. Laminates fabricated via OOA processing exhibited properties comparable to that of autoclave cured composites. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43984.

INTRODUCTION

The Out-of-Autoclave (OOA) process, specifically a vacuum bag only OOA process, is a low cost alternative to conventional autoclave manufacturing of composite components.[1,2] The OOA process requires low capital investment and provides greater design flexibility for the production of large structural components with complex geometries. The part size is no longer limited by the size of the autoclave.[3] On the other hand, autoclave pressures are high enough to suppress void formation. OOA cure processes can lead to higher void contents as the part is cured under atmospheric pressures.[4] Proper control of the prepreg breathing mechanism is required to produce void free parts. OOA prepregs feature dry, relatively permeable areas that allow gas evacuation when vacuum is applied for consolidation of layup. These areas consist of macro-porosity between plies and around the reinforcement architecture, or micro-porosity inside the tows, between individual fibers. During the cure cycle, once the melting point of the resin is reached, these pores are filled by the matrix material to produce a void free structure.[5]

The relationship between porosity and reduced mechanical properties is well established.[6] In autoclave processing, high working pressures combined with vacuum evacuation gives rise to a high pressure differential that enhances removal of entrapped air. OOA processes generally use atmospheric pressure and, therefore, venting entrapped air from the laminate stack is a key concern. Entrapped gasses need to be removed through engineered pathways provided within the prepreg.[7] The material must also exhibit low viscosity before cure and low outgassing during cure.

OOA processes using carbon/epoxy prepregs have been used to fabricate large and complex composite structures such as the Boeing Wing Spar, Advanced Composite Cargo Aircraft, and the Ares V interstage and payload shroud.[8,9] OOA processing of Bismaleimide (BMI)-based composite systems is relatively new.[10] BMIs are used in the aerospace industry due to higher service temperatures compared to epoxy systems, and better processing behavior compared to polyimides. BMIs also exhibit good tack and drape, and an epoxy-like addition cure mechanism. In addition, they possess desirable properties such as high tensile strength, corrosion and chemical resistance, and good hot-wet performance.[11] OOA BMI laminates were manufactured as a part of the NASA CoEx (Composite for Exploration) project using prepreg from Renegade Materials Corporation,
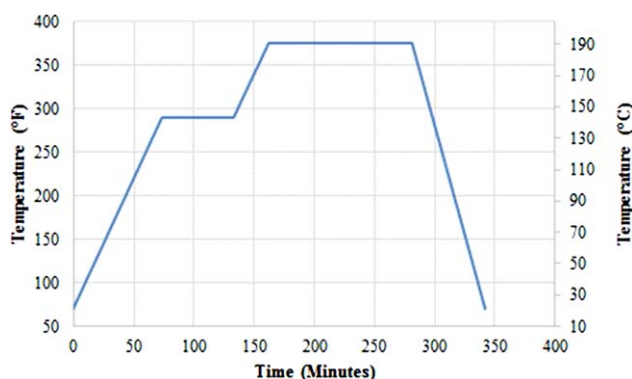
Figure 1. Manufacturer recommended cure cycle. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Tencate and Stratton Composite Solutions.[12] Sandwich composites made with BMI OOA prepregs were also evaluated as a part of CoEx.[13] Due to a limitation in tooling, the cure temperature in was limited to 350 °F (177 °C). The manufactured laminates had poor consolidation, high voids, and also a high glass transition temperature.

Cure conditions affect mechanical properties of thermosetting resins and composites. Mechanical properties and glass transition temperature of composite laminates can be improved by incorporating a post cure cycle. Rise in post cure temperature has been shown to increase degree of cure, glass transition temperature, and mechanical strength of carbon fiber/epoxy composite systems.[14] Post curing also increases the crosslink density in a composite. A high crosslink density leads to high glass transition temperatures because of stiffer polymer chains. On the other hand high crosslink densities also cause matrix embrittlement. At higher degrees of cure and crosslink densities, the glass transition temperature of BMI-based systems can approach 300 °C. He et al.[15] investigated the effect of post cure cycles on IM7/5250-4 composites. It was found that glass transition temperature increased with post-cure temperature. Flexure properties and mode II fracture toughness were also enhanced for composites post cured at 425 °F (218 °C), as compared with those post cured at (375 °F) 191 °C or at 475 °F (246 °C).

In the current work, BMI composite laminates are manufactured using OOA process. First, the quality of composite panels manufactured using the OOA prepreg was evaluated using void

content tests. Edge bleeding was used to evacuate entrapped air within the laminate stack. The void content of manufactured laminates was evaluated using acid digestion. Once the laminate quality was established, the effect of varying base cure time on mechanical properties of green composite panels was evaluated using short beam shear (SBS) tests. The influence of post cure conditions on glass transition temperature and mechanical strength of BMI composite panels was studied using Thermo-mechanical analysis (TMA) and SBS tests, respectively. Using these results, a composite panel was manufactured and its properties were compared with an autoclave cured specimen. OOA fabricated composited exhibited properties similar to autoclave cured parts.

MATERIALS

Composite laminates were manufactured using IM7G/AR4550 BMI unidirectional prepreg system (Aldila Composite Materials). AR4550 is a toughened BMI resin system, ideal for OOA curing. The unidirectional prepreg contains 35% resin by weight with a prepreg areal weight of 304.8 g/m^2. It has low tack compared to epoxy prepregs and has a shelf life of 2 weeks. To bleed entrapped air from the edge of a laminate stack, a light weight 54 gsm leno glass cloth and Vac-Pak EB1590 edge bleeder were used. EB1590 is an open weave, 600 °F (315 °C), high tensile strength Teflon-coated lightweight fiberglass material suitable for edge breathing. The Teflon coating ensures easy release from the composite laminate. The manufacturer recommended cure cycle is shown in Figure 1. The temperature is first raised to 290 °F (143.3 °C) for 1 h, in order to enhance the mobility of reacting groups and then raised to the required base cure temperature.

MANUFACTURING

Laminates were manufactured using an OOA prepreg process. The details of configuration and dimensions of the laminates manufactured are shown in Table I.

An aluminum mold was cleaned and covered with an Ethylene Tetrafluoroethylene (ETFE) release film. Prepreg layers were cut to size and placed on the mold until the laminate stack had the required number of layers. EB1590, a Teflon coated glass fabric, was used as an edge bleeder to remove entrapped air from the laminate stack. A layer of N10 breather (Airtech) was used for

Table I. Laminates Manufactured in the Study

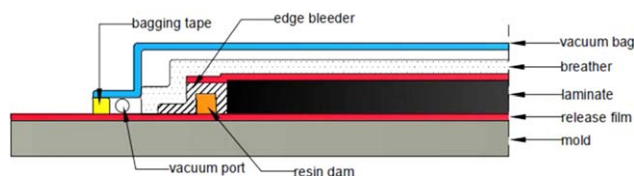| Number of laminates	Layup	Dimensions	Test
4	[0°/90°/+45°/−45°]$_{2s}$	6 × 6 × 0.076 in.^{3} (152.4 × 152.4 × 1.9 mm^3)	Porosity analysis
11	[0°/90°/+45°/−45°]$_{3s}$	12 × 12 × 0.12 in.^{3} (304.8 × 304.8 × 3.02 mm^3)	Base cure study
1	[0°/90°/+45°/−45°]$_{3s}$	12 × 12 × 0.12 in.^{3} (304.8 × 304.8 × 3.02 mm^3)	Post cure study
1	[0°]$_8$	12 × 12 × 0.039 in.^{3} (304.8 × 304.8 × 1 mm^3)	Comparison with autoclave cured laminates

Figure 2. OOA process bagging assembly. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
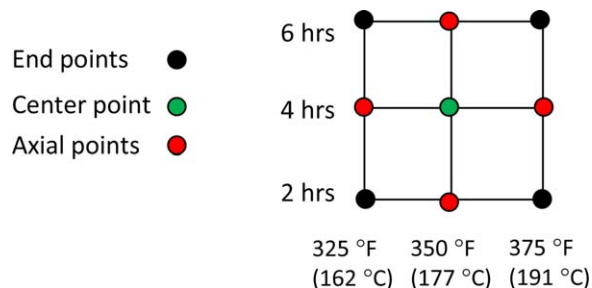

Figure 3. Face centered central composite design. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

even distribution of vacuum. The entire setup was sealed using a vacuum bag. The vacuum pressure was 28 in. Hg. Debulking was performed every four layers, for a duration of 30 min, to remove the entrapped air from the laminate stack and ensure good prepreg compaction. The bagging scheme is shown in Figure 2. A final warm debulk was performed at 121 °F (50 °C) for 1 h. The laminate was then sealed using a double bagging scheme and cured in an oven. The base cure options used in this study are shown in Table II. Test specimens were cut from the manufactured laminate for mechanical testing.

For the study on the effect of post-cure conditions, a single 24 ply laminate with orientation, $[0°/90°/+45°/-45°]_{3s}$, was manufactured using the base cure cycle, 375 °F (191 °C)/2 h. Test samples were extracted and post cured according to the post cure options in Table II.

METHODOLOGY

Porosity Study

Efficient air bleeding is required in order to obtain a relatively void free composite panel. Removal of entrapped air is an important part of the OOA process as working pressure differential is not high enough, compared to autoclave processing. Two bleeding techniques were evaluated—iber glass cloth edge bleeder and Vac-Pak EB1590. 16 ply, Quasi-isotropic laminates with layup, $[0°/90°/+45°/-45°]_{2s}$, were used. The manufacturer recommended base cure cycle (Figure 1) was used for all bleeder evaluation panels. Each panel was tested for void content by acid digestion according to ASTM D3171. Five samples (25.4 × 25.4 mm^2) were cut from the composite panels and density measured by water displacement according to ASTM D792. The samples were dissolved in concentrated sulfuric acid and the

resin was oxidized using hydrogen peroxide. The fibers were separated and weighed.

Influence of Base Cure Conditions

Properties such as chain length and degree of cure are influenced by the cure history of a polymer.[16] These properties have a direct impact on its mechanical properties. Low green strength (strength before post cure) can also result in droop during freestanding post cure.

Experimental Design. The influence of base cure cycles on mechanical properties of the composite laminate was evaluated utilizing experimental data via the design of experiments (DOE) approach. Interlaminar shear strength (ILSS) was the response variable. The effect of two factors were investigated, namely cure temperature and curing time. The experimental factors referred in DOE nomenclature are input variables, set at predetermined levels. A central composite design, rather than a full-factorial experiment was used (Figure 3). The Central composite design chosen for this study can be used to fit a second order model. The end points (−1 and +1 values in coded form) of the central composite design were temperatures of 325 °F (162 °C) and 375 °F (191 °C) and cure times of 2 and 6 h. A face centered central composite (CCF) design was selected with axial points corresponding to 350 °F (177 °C)/4 h, 375 °F (191 °C)/4 h, 350 °F (177 °C)/2 h, and 375 °F (191 °C)/6 h. The center point (0 in coded form), 350 °F (177 °C)/4 h was replicated thrice.

Short Beam Shear Test. Green strength (strength before post cure) of the composite panels was evaluated using SBS test. Eleven composite panels, according to the central composite design, were manufactured. Each laminate had 24 plies arranged in quasi isotropic stacking sequence $[0°/90°/+45°/-45°]_{3s}$. Five samples, measuring 38.1 × 7.62 × 3.02 mm^3, were extracted from each panel. The test was performed on an Instron 5985 machine at room temperature. Span to thickness ratio was 3:1. Samples were loaded under three point bending at a machine crosshead speed of 0.05 in. per minute (1.27 mm/min.).

Influence of Post Cure Conditions

Glass Transition Temperature. The glass transition temperature was evaluated using TMA. A sample of size 3 × 3 mm^2 was cut from the laminate and placed in the thermomechanical analyzer (Perkins-Elmer). A sample of size 3 × 3 × 3.02 mm^3 was cut from the laminate and heated to 572 °F (300 °C) at a rate of 9 °F (5 °C) per minute. The glass transition temperature is the

Table II. Cure Conditions

| Base cure options | | Post cure options | |
Temperature (°F/°C)	Time (h)	Temperature (°F/°C)	Time (h)
325 (162)	2	425 (218)	2
325 (162)	4	425 (218)	4
325 (162)	6	450 (232)	2
350 (177)	2	450 (232)	4
350 (177)	4	475 (246)	2
350 (177)	6	475 (246)	4
375 (191)	2	500 (260)	2
375 (191)	4	500 (260)	4
375 (191)	6		

Table III. Evaluation of Bleeders

Panels	Bleeder	Debulking	Mean void content (%)
1	Fiberglass cloth	Yes	0.78 ± 0.28
2	Fiberglass cloth	No	3.85 ± 1.19
3	EB1590	Yes	0.31 ± 0.13
4	EB1590	No	1.44 ± 0.65

temperature at which a second order transition in the rate of expansion of the sample was observed.

Mechanical Testing. The delamination resistance of the composite samples was evaluated using SBS, using the same experimental conditions as mentioned earlier. Three samples were tested for each post cure option. The results of mechanical testing at room temperature were evaluated using the design of experiments approach. A full factorial design was used. Post cure temperature and duration were the input factors. The factor, "post-cure temperature" had four levels, that is, 425, 450, 475, and 500 °F (218,232, 246, and 260 °C). The other factor, "post cure time" (duration) had two levels, 2 h and 4 h.

RESULTS AND DISCUSSION

Porosity Study

Both bleeder materials, fiberglass cloth and EB1590, were capable of producing low void contents in the manufactured laminate (Table III). Debulking every four layers resulted in a decrease in void contents. Both the fiberglass cloth as well as the EB1590 were capable of ensuring <1% void contents which is a requirement for aerospace composites. The Teflon coating in EB1590 offers easy release from the cured laminate compared to the 54 gsm fiberglass cloth. To ensure ease of processing as well as high laminate quality, EB1590 was selected. Therefore, laminates used to evaluate variations in the base cure and post cure cycles, were manufactured using the Vak-Pak EB1590 bleeder and a debulk cycle.

Influence of Base Cure Conditions

A change in cure cycles results has a significant effect on matrix dominated behavior such as interlaminar shear strength. Previous studies on cure kinetics of BMI-based thermoset systems have demonstrated that the dependence of reaction rate on cure conditions can be described by an nth order reaction model[17]:

$$\frac{d\alpha}{dt} = A \ \exp\left(\frac{-E}{RT}\right) \ (1-\alpha)^n \tag{1}$$

where α is the degree of cure, $d\alpha/dt$ is the reaction rate, E is energy of activation, T is the temperature, R is the universal gas constant, n is the reaction order, and A is a constant. The rate of reaction increases with an increase in temperature. The degree of cure of the composite system is expected to increase with an increase in the cure temperature as well as the cure duration. This behavior should be reflected in macroscopic properties such as ILSS, which are resin dominated.

Short Beam Shear Test. Results of short beam shear tests are shown in Figure 4. Failure was due to ply cracking in the central layers of the specimen, which is an acceptable mode of failure
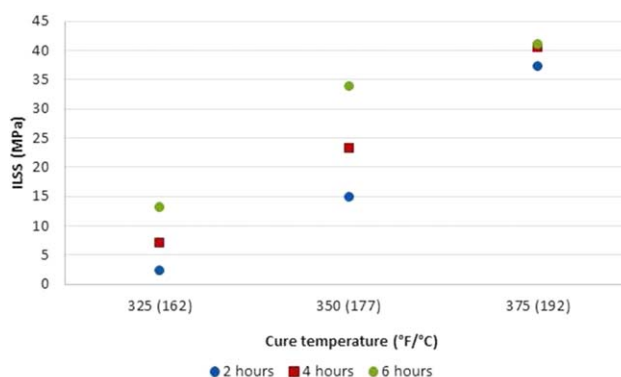

Figure 4. Effect of cure conditions on ILSS. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

according to ASTM D2344. On continued application of load, delamination of other layers was observed. Low ILSS of samples cured at 325 °F (162 °C) can be a result of low degree of cure at the central layers of the laminate. In general, a rise in base cure temperature and time resulted in an increase in short beam shear strength (Figure 4), which agrees with the hypothesis mentioned earlier.

In order to quantify the effect of cure temperature and time on ILSS, test results were evaluated using Analysis of Variance (ANOVA). In the current study, the null hypothesis is, "A change in factor levels, cure temperature and time, does not produce a significant change in the response variable." If the p-value, calculated using analysis of variance (ANOVA), is less than a pre-determined significance level, the null hypothesis can be rejected, concluding that: a change in base cure temperature and time results in a statistically significant change in laminate mechanical properties. In the current study, a significance level of 0.05 was selected. The p-value for model is 0.001 which indicates that the model is significant and the null hypothesis can be rejected (Table IV). In addition to testing model significance, the statistical significance of the two factors, cure temperature and curing time, were tested, specifically looking at the linear and quadratic effects of those variables on ILSS.

The p-values of the square terms as well as the interaction terms are greater than 0.05. Therefore, they do not have a significant effect on the response, ILSS, and can be rejected. Since the p-values of the linear terms (of both temperature and time) are less than 0.05, their effects are statically significant. Sample ILSS is affected by both base cure time and temperature.

A regression model was fit, in order to study how the ILSS changes in response to a change in base cure temperature and time. A regression model is an empirical relation which relates the factors to the responses considered in the experiment. The response, ILSS can be related to the factors as a second order quadratic equation,

$$\begin{aligned} ILSS = \beta_0 + \beta_1\left(Time\right) + \beta_2\left(Temperature\right) + \beta_3\left(Time^2\right) \\ + \beta_4\left(Temperature^2\right) + \beta_5\left(Time * Temperature\right) + \varepsilon \end{aligned} \tag{2}$$

Where the terms labeled "β" represents coefficients to be determined and ε is the error. Since the effects of square and interaction terms are negligible, their coefficients were set to zero. The variation of ILSS with time and temperature is shown in eq.

Table IV. ANOVA Table for the Effect of Base Cure Conditions on ILSS

Term	Degrees of freedom	Adjusted sum of squares	Adjusted mean squares	F	p
Model	5	1748.58	349.71	34.75	0.001
Linear	2	1735.08	867.54	86.19	0.000
Temperature (°F)	1	1547.00	1547.00	153.70	0.000
Time (h)	1	188.08	188.08	18.69	0.008
Square	2	0.42	0.21	0.02	0.979
Temperature (°F) * Temperature (°F)	1	0.10	0.01	0.01	0.979
Time (h) * Time (h)	1	0.40	0.04	0.04	0.924
Interaction	1	13.08	13.08	1.30	0.306
Temperature (°F) * Time (h)	1	13.08	1.30	1.30	0.306
Error	5	50.33	10.07		
Lack-of-Fit	3	49.83	16.61	67.39	0.015
Pure Error	2	0.49	0.24		
Total	10	1798.90			

(2). The response surface plot corresponding to the regression equation is shown in Figure 5.

$$ILSS = -212.3 + 0.6423\,(Temperature) + 2.799\,(Time) \qquad (3)$$

Where ILSS is interlaminar shear strength, Temperature is expressed in °F and Time is expressed in hours.

As seen in Figure 5, the delamination resistance increases with a rise in curing temperature as well as time. A rise in cure temperature and time can result in an improvement in the degree of cure, resulting in a better bond between the fiber and matrix. Accordingly, the laminate fabricated using a cure condition of 375 °F (191 °C)/6 h exhibited the greatest ILSS of 41.06 MPa. A 4.67 °F (2.59 °C) change in cure temperature is capable of producing a similar effect on delamination resistance, compared to a 1 h change in cure time.
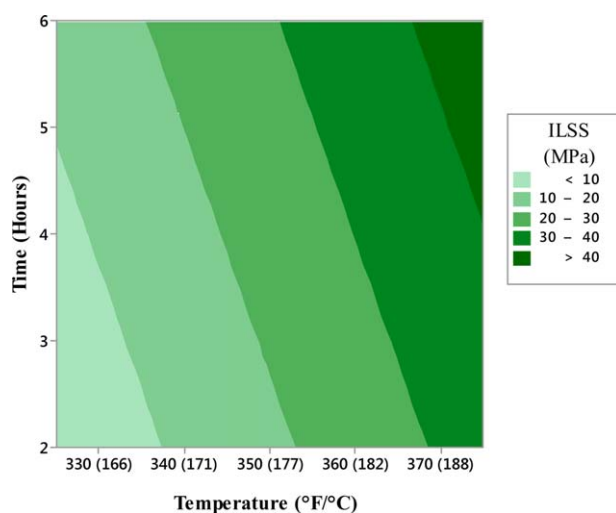
Verification of Results. Lowering base cure temperatures can reduce tooling costs. The results of the response surface design show that using a base cure cycle of 360 °F (182.2 °C)/6 h can result in a laminate of high quality compared to the manufacturer recommended cure cycle of 375 °F (191 °C)/2 h. For the purpose of verification, a composite panel was manufactured using a cure cycle of 360 °F (182.2 °C)/6 h. The sample thickness was 2.99 mm, void content was 0.83% and the density was 1.56 g/cm³. Five samples were removed and their ILSS was measured by short beam shear. The average ILSS was calculated to be 38.1 MPa. This compares well with the observed value of ILSS (37.43 MPa) at the manufacturer recommended cure cycle.

Influence of Post Cure Conditions

The purpose of post curing a composite is twofold: to improve the degree of cure and to increase the crosslink density. Results from prior Fourier transform infrared spectroscopy of BMI systems have indicated the presence of multiple reactions during cure.[18–20] In early stages of cure, the chain extension reactions dominate.[21] During later stages of cure, the reaction mechanism

Figure 5. Response surface plot of the influence of base cure temperature and time on ILSS. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
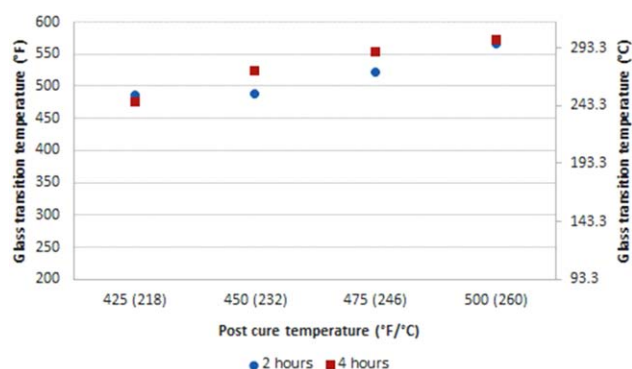

Figure 6. Variation of glass transition temperature with post cure conditions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
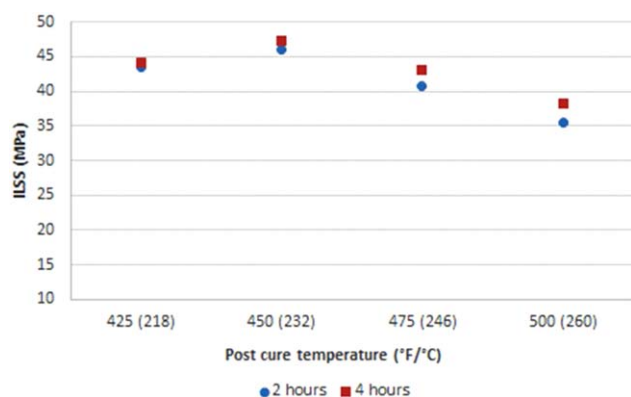
Figure 7. Variation of ILSS with post cure conditions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

is dominated by crosslinking. The crosslinking reaction occurs mainly above 180 °C.

Glass Transition Temperature. All post cure cycles were found to be capable of producing a composite that has a T_g higher than conventional epoxies. Glass transition temperatures of post cured samples increased with increase in post cure temperature in a linear fashion (Figure 6). Increase in the post cure time also lead to a rise in T_g. This is a direct result of an increase in the crosslink density between polymer chains. Maximum T_g was exhibited by samples post cured at 500 °F (260 °C) for 4 h.

Mechanical Testing. Using short beam shear tests, ILSS of green composite specimens was found to be 38.1 MPa. All post cure options resulted in an increase in ILSS of the composite specimens, which is due to the increase in degree of cure and crosslink density (Figure 7). Samples failed by the means of matrix cracking in the central layers of the specimens.

The results of mechanical testing, evaluated using two-way ANOVA, are shown in Table V. A significance level of 0.05 was used for the analysis. The p-value for model significance was 0.004. Since this is less than 0.05, a change in post cure temperature and time has a significant effect on the properties of the manufactured laminate.

The effect of interaction between the factors is considered statistically insignificant, because the p-value is 0.849 (>0.05). The p-value of the temperature of the effect of temperature was 0.001 which indicates that this factor has a significant effect on the response. The effect of post cure time is less significant (p-value = 0.069) than the post cure temperature. The factor, post cure time, was retained in the regression model [eq. (3)] because of its proximity to the cutoff value of 0.05. The factor ILSS, shows a dependence of the form,

$$ILSS = -497 + 1.151(\text{Time}) + 2.421(\text{Temperature}) - 0.002723(\text{Temperature})^2 \tag{4}$$

Where ILSS is interlaminar shear strength, Temperature is expressed in °F and Time is expressed in hours.

According to the regression equation, obtained by least squares fitting, maximum ILSS is obtained after post cure conditions of 444.5 °F (229 °C)/4 h. The increase in ILSS is due to improved degree of cure and increased crosslinking in the polymer chains. When samples are post cured at temperatures higher than 444.5 °F (229 °C), the mechanical properties begin to degrade. Results for literature indicate that the degree of crosslinking increases with an increase in post cure temperature.[15] The initial increase in ILSS can be explained because of an increase in matrix rigidity due to increased crosslink density. On the other hand, excessive crosslink densities can lead to matrix embrittlement which

Table V. ANOVA Table for the Effect of Post Cure Conditions on ILSS

Term	Degrees of freedom	Adjusted sum of squares	Adjusted mean squares	F	p
Model	7	291.42	41.63	4.97	0.004
Temperature (°F)	3	252.93	84.30	10.07	0.001
Time (h)	1	31.82	31.82	3.80	0.069
Interaction	3	6.67	2.22	0.27	0.849
Error	16	133.9	188.08		
Total	23	425.32			

Table VI. Comparison of Autoclave and OOA Cured Composite Laminates

	OOA cured	Autoclave cured[a] (85 psi)
Cure cycle	290 °F (143 °C) for 2 h;	290 °F (143 °C) for 1 h;
	375 °F (191 °C) for 2 h	310 °F (154 °C) for 1 h;
		375 °F (191 °C) for 2 h
Post cure	450 °F (191 °C) for 4 h	410 °F (210 °C) for 2 h
Layup	$[0°]_8$	$[0°]_8$
ILSS	143.54 MPa	139.96 MPa

[a] Source-manufacturer data sheet.

is associated with a reduction in resin dominated mechanical properties. The mechanical properties of the BMI composite can be improved by post curing but they will drop if the temperature is too high. Similar reduction in resin dominated properties such as mode II fracture toughness and flexure strength was reported by He *et al.*[15] for autoclave cured Cycom IM7/5250-4 composites.

COMPARISON WITH AUTOCLAVE CURED COMPOSITES

Unidirectional (8 ply) composite laminates were manufactured via OOA process. The laminates were cured at 375 °F (192 °C)/ 2 h followed by a free standing post cure at 450 °F (232 °C) for 4 h. The samples showed no warping on post cure. Test samples were cut and the ILSS was measured according to ASTM D2344. A span to thickness ratio of 4:1 was used. The loading rate was 1.27 mm per minute. The ILSS of the manufactured laminates was 143.54 MPa. The mechanical properties of the fabricated laminate were compared with those of autoclave cured composites (data provided by manufacturer), using the same prepreg. The properties of OOA cured laminate are comparable with the properties of the specimens fabricated in an autoclave under 85 psi (Table VI).

CONCLUSIONS

Laboratory scale Carbon/Bismaleimide composite laminates were manufactured successfully using the OOA process. Edge bleeding with debulking every four layers was found to be successful in producing parts with low void contents (<1%). The effect of cure cycle variations on mechanical and thermal properties of the OOA cured BMI composite laminates was evaluated. Low base cure temperatures produced parts with relatively low mechanical strengths. Maximum interlaminar shear strengths were obtained when parts were cured at 375 °F (191 °C) for 6 h. Base cure cycles involving lower temperatures and cure times resulted in a drop in ILSS. A base cure cycle of 360 °F (182 °C)/6 h can result in a laminate having ILSS comparable to one manufactured using the recommended cure cycle of 375 °F (191 °C)/2 h.

The variation of T_g and ILSS with post-cure conditions was evaluated. Samples were post cured at temperatures between 425 °F (218 °C) and 500° F (260 °C). Post cure times of 2 h and 4 h were evaluated. An increase in post cure temperature as well as post cure time led to a rise in T_g. The measured T_g was sufficient to replace conventional epoxy resins in high temperature applications. The interlaminar shear strength was affected by post cure temperature. Post curing up to a temperature of 444.5 °F (229.1 °C) results in an increase in ILSS. The mechanical properties degrade at higher post cure temperatures, possibly due to thermal degradation and excessive crosslinking. The effect of post cure time is less significant compared to the effect of post cure temperature. Based on the results of experimental testing it was found that maximum ILSS can be obtained in samples post cured at 444.5 °F (229.1 °C) for 4 h. Test samples were manufactured at 375 °F (191 °C)/2 h and post cured at 450 °F (232 °C)/4 h. Their properties compared well with those of an autoclave cured composite.

REFERENCES

1. Ridgard, C. In Proceedings of the International SAMPE Symposium and Exhibition; Seattle, WA, May 17–20, **2010**; pp 1–18.

2. Sakota, Z.; Thomas Hahn, H.; Lackman, L.; Bullen, G. In Proceedings of the International SAMPE Symposium and Exhibition; Long Beach, CA, April 30–May 4, **2006**; pp 1–13.

3. Centea, T.; Gunenfelder, L.; Nutt, S. *Compos. Part A: Appl. Sci. Manuf.* **2015**, *70*, 132.

4. Grunenfelder, L.; Nutt, S. *Compos. Sci. Technol.* **2010**, *70*, 2304.

5. Centea, T.; Hubert, P. *Compos. Sci. Technol.* **2011**, *71*, 593.

6. Costa, M. L.; Almeida, S. F.; Rezende, M. C. *Compos. Sci. Technol.* **2001**, *61*, 2101.

7. Levy, A.; Kratz, J.; Hubert, P. *Compos. Part A: Appl. Sci. Manuf.* **2015**, *68*, 365.

8. Marsh, G. *Reinforced Plast.* **2012**, *56*, 20.

9. Sutter, J.; Kenner, W.; Pelham, L.; Miller, S.; Polis, D.; Nailadi, C.; Hou, T.; Quade, D.; Lerch, B.; Lort, R.; Zimmerman, T.; Walker, J.; Fikes, J. In Proceedings of the SAMPE Fall Technical Conference; Salt Lake City UT, October 11–14, **2010**; pp 1–15.

10. Stratton, R.; Repecka, L. In Proceedings of the International SAMPE Technical Conference; Long Beach, CA, May 24–26, **2011**; pp 1–10.

11. Stenzenberger, H.; Herzog, M.; Koenig, P.; Roemer, W.; Breitigam, W. In Proceedings of the International SAMPE Symposium and Exhibition; Reno, NV, May 8–11, **1989**; pp 1877–1888.

12. Hastings, W.; Pelham, L.; Miller, S.; Sutter, J.; Martin, R.; McCorkle, L.; Scheiman, D. In Proceedings of the International SAMPE Technical Conference; Long Beach, CA, May 6–9, **2013**; pp 1347–1360.

13. Hou, T.; Miller, S.; Williams, T.; Sutter, J. *J. Reinforced Plast. Compos.* **1014**, *33*, 137.

14. Vo, T.; Vora, K.; Minaie, B. *J. Appl. Polym. Sci.* **2013**, *130*, 3090.

15. He, Y.; Zhong, Y.; Zhou, J. In Proceedings of the International SAMPE Technical Conference; San Antonio, TX, October 20–24, **1998**; pp 226–233.

16. Painter, P.; Colemon, M. In Fundamentals of Polymer Science: An Introductory Text; CRC Press: New York, **1998**.

17. Guo, Z.; Du, S.; Zhang, B.; Wu, Z. *J. Appl. Polym. Sci.* **2005**, *97*, 2238.

18. Tungare, A.; Martin, G. *J. Appl. Polym. Sci.* **1992**, *46*, 1125.

19. Mijović, J.; Andjelić, S. *Macromolecules* **1996**, *29*, 239.

20. Morgan, R.; Shin, E.; Rosenberg, B.; Jurek, A. *Polymer* **1997**, *38*, 639.

21. Boey, F.; Song, X.; Yue, C.; Zhao, Q. *J. Polym. Sci. Part A: Polym. Chem.* **2000**, *38*, 907.