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Fabrication of out-of-autoclave bismaleimide based composite laminates with embedded fiber optic sensors

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FABRICATION OF OUT-OF-AUTOCLAVE BISMALEIMIDE BASED COMPOSITE LAMINATES WITH EMBEDDED FIBER OPTIC SENSORS

by

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ABSTRACT

Composites are becoming the material of choice in applications where weight savings are critical, like aerospace structures. The common composites used are- Carbon/Epoxy and Carbon/Bismaleimide (BMI). BMI based systems are preferred in applications which involve operating temperatures higher than conventional epoxies. Carbon/BMI laminates are traditionally fabricated in an autoclave, which is associated with high operating costs. In this work, a low cost out of autoclave (OOA) process is evaluated. It is desirable to have BMI OOA prepreg systems cure at reasonably low temperatures with sufficient degree of cure and green strength to maintain rigidity for subsequent freestanding post cure Carbon/BMI composite laminates are manufactured using an OOA compatible prepreg and the effect of varying base cure cycles on the green strength (strength before post cure) is investigated.

In aerospace structures, Carbon/BMI composites are used in high temperature applications. Fiber optic sensors are a compact non-intrusive means of structural health monitoring under these conditions. Optical fiber based sensors have many advantages like their compact size, resistance to corrosion, immunity from electromagnetic interference, and multiplexing capabilities. Embedded fiber optic sensors are used to study stresses developed during cure of carbon/BMI composite laminates. The same sensors are then used to measure strain developed in the composite on the application of mechanical loads.

ACKNOWLEDGMENTS

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1. INTRODUCTION

1.1. BISMALEIMIDES IN AEROSPACE APPLICATIONS

In structural applications, fiber-reinforced polymer matrix composites offer significant advantages over other materials because of their low density and high specific strength. These composites are especially attractive for use in the aerospace industry, where weight savings are critical. This reduction in weight can have substantial benefits in the form of lower fuel consumption, an increased load carrying capacity, or increased speed and maneuverability (Military airplanes).

The durability of composites used in aerospace components is critical. Some important material requirements are a high glass-transition temperature (T_g) resistance to environmental degradation, and good mechanical properties over a wide range of temperatures. Conventional epoxies used in aerospace industry are limited by an operating temperature of about 250 °F. Bismaleimide resins generally exhibit a glass transition temperature greater than 500 °F which make them ideal candidates for use in temperature range above conventional epoxy resins.

Figure 1.1 Structure of BMI resin, Ar- aromatic group

In industrial applications, processability of composites is a key concern. Polymers with structures like aromatic rings in the backbone, can have a high T_g, but can exhibit poor solubility in most organic solvents, high melting or softening points, and melt viscosities that are too high to allow their processing by conventional techniques.

Major advantages of BMIs are that they combine high temperature performance with an epoxy-like processing. They also have properties comparable to that of medium epoxies (MTE) and high toughness epoxies (HTE, Table 1.1) used in aerospace applications. This enables their use in aerospace grade composite structures.

Table 1.1 Comparison of properties of BMI with MTE and HTE*

Property	**5250-4 IM7/ BMI**	**977-3 G40-800 MTE**	**5276-1 G40-800 HTE**
Tensile properties			
Strength (MPa)	2827	2758	2827
Modulus (Gpa)	161	163	164
0 ° Compression strength			
RT (MPa)	1689	1689	1586
80 °C (MPa)	1586	1448	1310
Open Hole compression			
RT (MPa)	324	325	310
80 °C/wet (MPa)	283	283	348
125 °C/wet (MPa)	262	262	193
175 °C/wet (MPa)	241	NA	NA
Compression after impact	185-200		
Tg			
Dry °C	280	210	180
Wet °C	210	165	145

*Stenzenberger H.D., "Structural Adhesives:Developments in resins and primers, A.J. Kinloch ed.", book pp. 77-126 (1986)

1.2. COMPOSITE MANUFACTURING PROCESSES

Aerospace grade composites are traditionally cured in an autoclave under high pressures of around 100 psi. The composite layup is sealed by a vacuum bag and connected to a vacuum pump. This establishes a pressure differential which forces entrapped air out of the laminate stack and ensures high part quality. Traditionally, BMI composite laminates for aerospace applications were manufactured in an autoclave. The high pressure ensures good compaction of the laminate and reduces void contents.

It is highly desirable to be able fabricate high quality composite parts without the use of an autoclave. OOA Vacuum bag only process offers one viable solution. OOA processes offer design flexibility in the manufacture large structural composites with complex geometries as they are not limited by the size of an autoclave. They also offer significant savings in operating costs, process time, capital investment and energy efficiency.

Acquiring and operating large autoclaves, required for traditional manufacturing of high performance composites can be expensive. Moreover, the part size is limited by the size of the autoclave. Use of OOA processing can also result in reduced core crush and core stabilization (sandwich structures), use of low cost tooling and production flexibility. In OOA process, BMI composite laminates are manufactured at atmospheric pressure in an oven. The major downside of OOA processing is that removal of voids is not as efficient as in an autoclave, due to lower pressure differential during cure. Voids content in a composite laminate can result in reduced mechanical properties.

1.3. CURING OF BMI BASED RESIN SYSTEMS

Two step cure cycles are generally used to cure composite laminates. A dwell time is incorporated at the temperature where the resin shows maximum viscosity. This is done in order to increase the mobility of reacting groups and ensure more uniform curing.

The temperature is then ramped up to the actual cure temperature (base cure). It is held for a time based on thickness of laminate and type of resin. Epoxies and BMIs generally have a 2 hour cure time (Figure 1.2). Post curing of composite laminates can result in an increase in degree of cure due to a decrease in the percentage of unreacted monomer remaining in the sample. If a composite contains regions of insufficient degree of cure, it can be susceptible to creep. A free standing post cure is performed, after the base cure cycle, to avoid these issues.

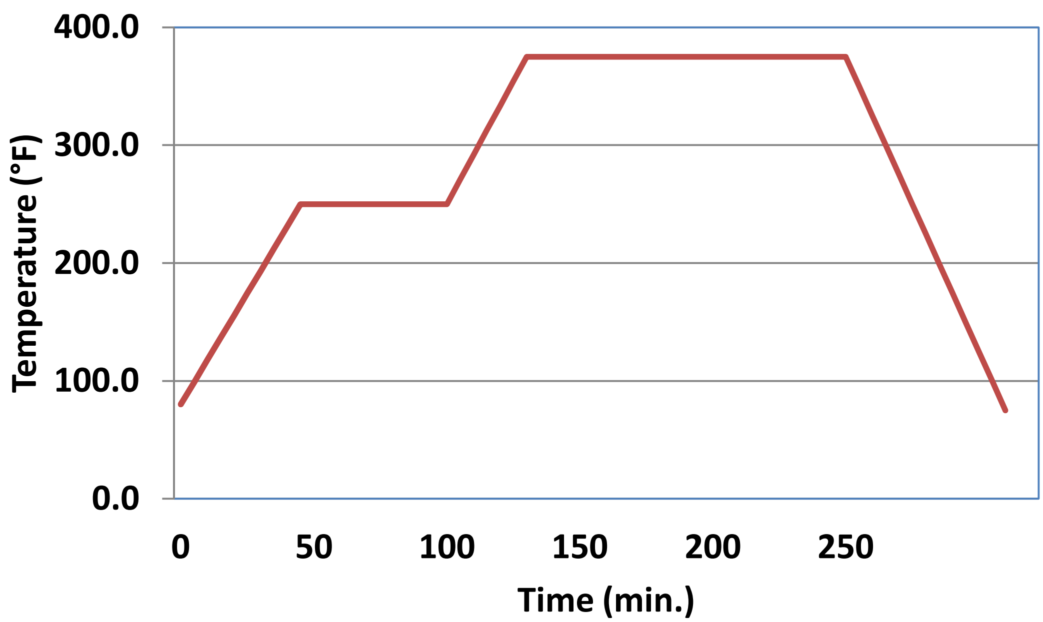

Figure 1.2 Cure cycle for OOA-BMI prepreg

1.4. CONTRIBUTION OF THIS WORK

Conventionally BMI systems have been manufactured using an autoclave. A wealth of literature is available, related to autoclave processing of BMI composites, but OOA processing of BMI is relatively new. The work presented in this thesis will focus on processing carbon fiber/BMI composites at atmospheric pressure in a conventional oven.

The first part will focus on optimizing the base cure cycle of BMI prepregs in order to obtain sufficient green strength after cure. The second part will investigate structural health monitoring of carbon fiber/BMI composites using fiber optic sensors.

I. PAPER

INFLUENCE OF CURE CONDITIONS ON PERFORMANCE OF OUT-OF-AUTOCLAVE CURED CARBON/BISMALEIMIDE COMPOSITES

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ABSTRACT

Bismaleimide (BMI) resins are a class of polyimides used in high-performance structural composites that require superior toughness and high-temperature resistance. Out-of-autoclave (OOA) processing of composites offers several key benefits compared to autoclave processing such as lower manufacturing cost resulting from a lower capital cost and lower energy consumption. Since few composite manufacturers have large, high-temperature autoclaves, OOA processing of BMI can broaden the use of these materials and expand the high-temperature composite parts supplier base. It is desirable to have BMI OOA prepreg systems cure at reasonably low temperatures with sufficient degree of cure and green strength to maintain rigidity for subsequent freestanding post cure. In the present work, high-temperature composite laminates are manufactured using BMI OOA prepreg. Venting of entrapped air from the prepreg stack is evaluated. Composite panels are manufactured using the most efficient venting method and short beam shear strengths are measured. Free-standing post cures are performed and the panels are monitored for droop. It was found that composite panels cured at 360 for six hours can produce properties similar to that of composites cured using the recommended cure cycle.

1. INTRODUCTION

High performance composites in aerospace applications can be exposed to prolonged and sometimes extreme service conditions. In aerospace applications, durability and reliability of composites is critical. A high glass transition temperature, good properties at elevated temperatures, and low susceptibility to environmental conditions is preferred [1]. Bismaleimides (BMI) are polyimides which exhibit good thermal stability, low water absorption, and high mechanical properties at high temperature. BMI resins provide better mechanical performance at elevated temperatures compared to conventional toughened epoxy resins. In addition, they possess desirable properties such as high tensile strength, corrosion and chemical resistance, and good hot-wet performance [2].

Autoclave processing has been shown to provide low void content and high quality parts for aerospace applications but is associated with high costs. Out-of-Autoclave (OOA) processes have a potential to reduce capital and operating costs. Complex parts can be manufactured using OOA processes in a simple, cost-effective manner. The major downside of OOA processing is the relatively higher void content (or porosity) [3]. Many researchers have investigated the effect of porosity on mechanical properties like compression [4], interlaminar shear [5, 6, 7], flexure strength [8] and tensile strength [9, 10]. When processing composites, entrapped air can be removed through the laminate by mass transport or momentum transport. In autoclave processing, high working pressures combined with vacuum evacuation gives rise to a high pressure differential that enhances removal of entrapped air by momentum transport [11]. 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 [12]. While substantial literature is available regarding autoclave cured BMI composites, limited work has been done to investigate out of autoclave oven cured BMI laminates.

Resin properties such as degree of cure and composite properties like residual stresses are affected by the cure cycle [2, 13, 14]. Cure conditions affect mechanical properties of thermosetting resins and composites [6, 15]. Certain mechanical properties and glass transition temperature of composite laminates can be improved by incorporating a post cure cycle [16]. Post curing has also been shown to decrease the percentage of unreacted monomer present in a composite [17]. Thermal warping can be induced in the laminate due to volumetric contraction of the resin or mismatch in coefficients of thermal expansion between the resin and fiber.

In the current work, air bleeding or venting of entrapped gasses within the laminate stack, is evaluated using three methods. The baseline method uses glass fabric as an edge bleeder. The second method uses Vac-Pak EB1590. In addition, a surface bleeder fabric, Trans-Textil C2003 is evaluated. A suitable bleeding process is selected, based on void content tests, which is used to manufacture laminates for mechanical testing and cure cycle evaluation. The effect of varying base cure time on mechanical properties of green composite panels (before post cure) is evaluated using short beam shear (SBS) tests. Thermal warping induced during free standing post cure is studied.

2. 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 fiber areal weight of 200 g/m^2. To bleed entrapped air from the edge of a laminate stack, a light weight 54 gsm leno glass cloth and a Vac-Pak EB1590 were used. EB1590 is an open weave, 600 °F, high tensile strength teflon-coated, and lightweight fiberglass material suitable for edge breathing. The teflon coating ensures easy release from the composite laminate. A third material, Trans-Textil C2003 was evaluated as a surface bleeder. The C2003 is a lightweight multilayer, flexible membrane system that acts as a resin barrier and was evaluated for its effectiveness in removing air from the surface of a laminate stack.

3. MANUFACTURING

Laminates were manufactured using an Out-of-Autoclave (OOA) or Vacuum Bag Only (VBO) process (Figure 1). Two sets of BMI composite laminates were fabricated. Six in. square 16 ply laminates were manufactured for void content tests and 16 in. square 24 ply laminates for cure cycle evaluation. Each laminate was laid up in a symmetric quasi-isotropic configuration [0°, 90°, +45°, -45°].

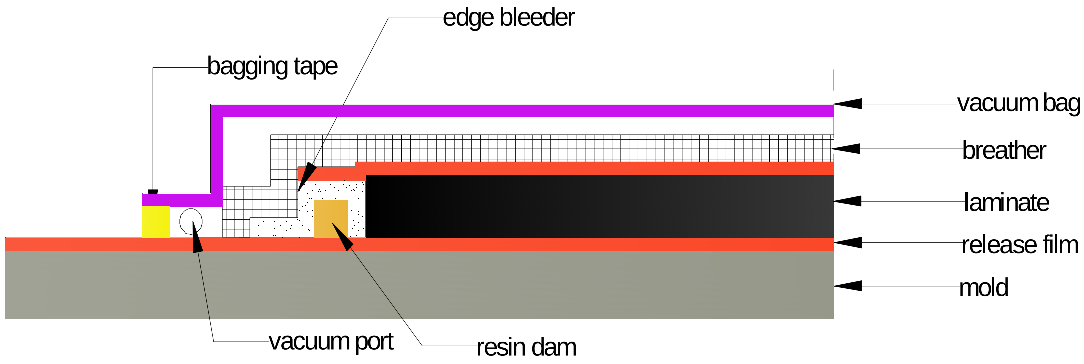

Figure 1. OOA Process Bagging Assembly with Edge Bleeder

In this study, debulking was performed every four layers for a duration of thirty minutes each to remove the entrapped air from the laminate stack and produce lower void contents in finished parts. A two-step cure cycle was used to cure the laminate. The temperature was first raised to 290 °F for one hour, in order to enhance the mobility of reacting groups. The laminate was then cured in an oven according to one of the cure cycle options listed in Table 1.

Table 1. Cure Cycle Options

Panel	**Temperature (°F)**	**Time (hours)**	**Panel**	**Temperature (°F)**	**Time (hours)**
1	325	2	7	350	2
2	375	2	8	350	6
3	325	6	9	350	4
4	375	6	10	350	4
5	325	4	11	350	4
6	375	4			

4. EXPERIMENTAL TESTING

4.1. BLEEDER EVALUATION

Thermogravimetric analysis (TGA) was performed to investigate outgassing of the BMI prepreg under cure conditions. The curing time of a small sample of BMI was found to be 9 minutes by dynamic DSC runs. A prepreg sample was placed in the platinum pan and a shortened cure cycle was simulated in a TA800 instrument. The percentage mass drop, during cure at 375°F, was found to be negligible (Figure 2). There is little evolution of volatiles during the cure process.

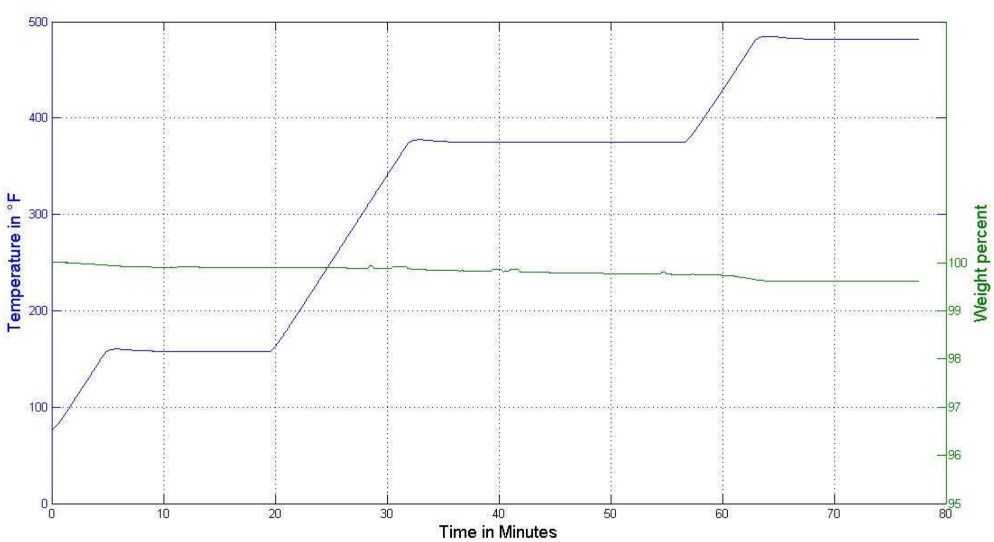

Figure 2. Mass loss of BMI prepreg under cure conditions

Removal of entrapped air is an important part of the OOA process as working pressure differential is not high enough (compared to autoclave processing) to remove entrapped air completely [11]. Efficient air bleeding is required in order to obtain a relatively void free composite panel. In the current study, three bleeding techniques were evaluated- fiber glass cloth edge bleeder, Vac-Pak EB1590 (Figure 3) edge bleeder and Trans-Textil C2003 (Figure 4) surface bleeder. Six 6-in. square, 16 layer quasi-isotropic laminates ($[0°, 90°, +45°, -45°]_{2s}$) were manufactured in order to evaluate bleeder selection. The manufacturer recommended base cure cycle of 375 °F for two hours was used for all bleeder evaluation panels.

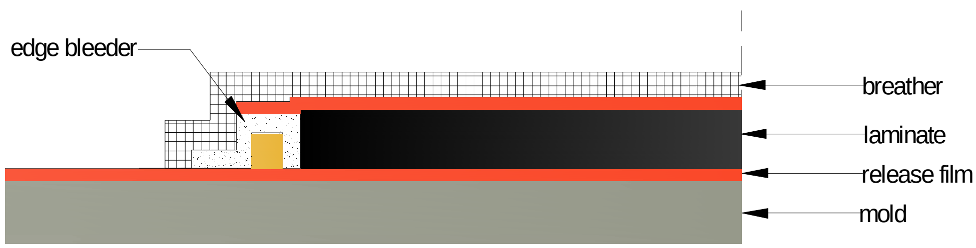

Figure 3. Edge Bleeding in OOA Process

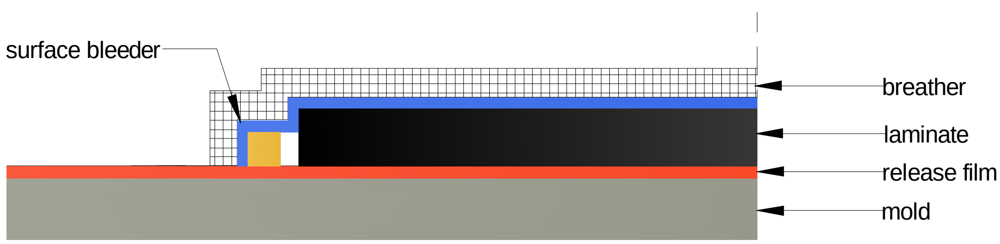

Figure 4. Surface Bleeding in OOA Process

Each panel was tested for void content by acid digestion according to ASTM D3171 [19]. Five, 1 in. square, samples 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. Void contents were measured using equations 1-3.

Table 2. Evaluation of bleeders

Panels	**Bleeder**	**Debulking**	**Void content (%)**
1	fiberglass cloth	Yes	0.78
2	fiberglass cloth	No	3.85
3	EB1590	Yes	0.31
4	EB1590	No	1.44
5	C2003	Yes	4.27
6	C2003	No	3.84

$$V_f = M_f \times \frac{density\ of\ sample}{density\ of\ fiber}, \tag{1}$$

$$V_r = M_r \times \frac{density\ of\ sample}{density\ of\ resin}, \tag{2}$$

$$V_v = 100 - V_r - V_f, \tag{3}$$

where, M_f= mass percent of fiber, M_r= mass percent of resin, V_f= volume percent of fiber, V_r= volume percent of resin, V_v = void content

Usage of baseline bleeder as well as EB1590, with debulking, resulted in the low void contents (Table 2). Vac-Pak EB1590 offers easy release from the laminate as compared to the 54 gsm glass cloth. Therefore, further laminates, used to evaluate variations in the cure cycle, were manufactured using the Vak-Pak EB1590 bleeder and a debulk cycle.

4.2. MECHANICAL TESTING

Green strength of the composite panels was evaluated using Short beam shear test. Eleven composite panels, measuring 16 in. square, were manufactured. Each laminate had 24 layers arranged in quasi isotropic stacking sequence $[0°, 90°, +45°, -45°]_{3S}$. Five 0.3 in. x 1.5 in. short beam shear (SBS) samples were extracted from each panel. The test was performed on an Instron 5985 machine at room temperature. A short span ratio of 3:1 was used to cause failure dominated by delamination [18].

The influence of cure cycle on delamination resistance was studied for a series of fabricated composite panels using the short beam shear test (Figure 5). Samples were loaded under three point bending on a loading span of 0.8 in., in accordance with ASTM D2344 [20]. A machine crosshead speed of 0.05 in. per minute was used to apply the load. Acceptable mode of failure in SBS is shear failure between the central layers of the specimen. The interlaminar shear strength (ILSS) was calculated using the following equation,

$$F^{sbs} = 0.75 \times \frac{P_m}{b \times d}, \tag{4}$$

Where P_m = peak load, b= sample width, d= depth of the specimen

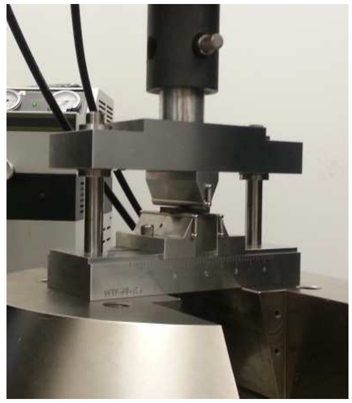

Figure 5. Short Beam Shear Test Setup

4.3. DROOP AFTER FREE-STANDING POST-CURE

Shrinkage of matrix around the reinforcing fibers gives rise to residual stresses. These stresses are capable of introducing dimensional changes in a laminate with low green strength. In the present work, droop during free standing post cure is studied. Test specimens measuring 14 in. x 10 in. were cut from panels manufactured, and placed between 1 in. wide aluminum mounting blocks (Figure 6). The panels were post-cured at 400 °F for two hours and droop was measured.

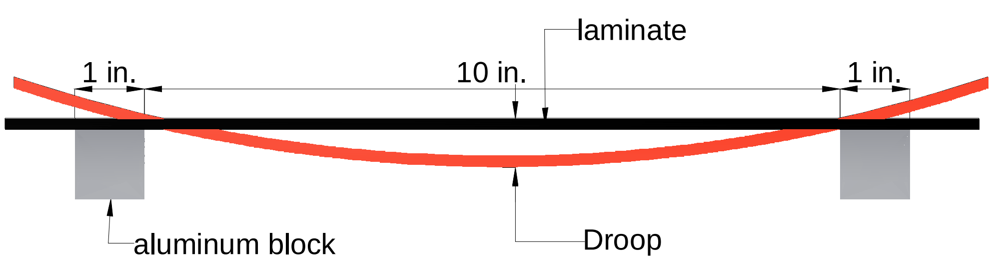

Figure 6. Droop under Free-Standing Post Cure

5. STATISTICAL ANALYSIS

Results from experiments were evaluated using statistical methods using Analysis of Variance technique. Interlaminar shear strength, flexure strength and droop were selected as the response variables. The control factors are input variables, set at predetermined levels. Two factors are involved, cure temperature and curing time. A face centered central composite design and the number of panels needed was determined based on that design. Central composite designs can be used to fit a first order model while providing information regarding contribution of second order terms. Using dynamic DSC scans, it was found that the onset of cure of the BMI resin is 290 °F. The extreme points of the central composite design were temperatures of 325 °F and 375 °F and cure times of 2 hours and 6 hours (Figure 7). The face centered cubic design was selected with axial points corresponding to 350 °F/2 hours, 350 °F/6 hours, 325 °F/2 hours and 375 °F/4 hours. The center point, 350 °F/4 hours was replicated thrice in order to determine whether first and second order terms have an effect on the response variables. Composite panels were manufactured as in Table 1.

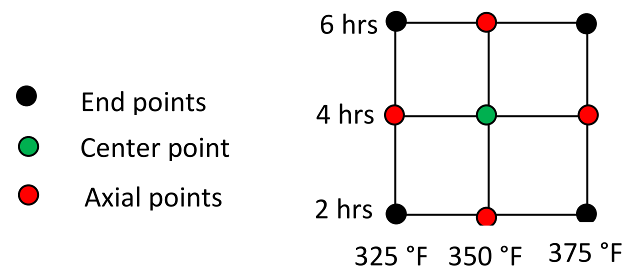

Figure 7. Face Centered Cubic Design

6. RESULTS AND DISCUSSION

Test results were evaluated using Minitab statistical software. 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 i.e. a change in factor levels results in a significant change in the response variable. In the current study, a significance level of 5% is used. Therefore, if $p<0.05$, a change in factor levels is significant.

Table 3. Table of Results

Panel	**Cure Temp. (°F)**	**Cure Time (hours)**	**ILSS (MPa)**	**Droop (mm)**
1	325	2	2.4	0.508
2	375	2	37.43	0.304
3	325	6	13.27	0.406
4	375	6	41.06	0.152
5	325	4	7.11	0.279
6	375	4	40.64	0.101
7	350	2	14.92	0.177
8	350	6	34.02	0.101
9	350	4	23.4	0.457
10	350	4	22.71	0.406
11	350	4	23.68	0.279
Significance of factors			0.001	0.612

6.1. MECHANICAL TESTING

Results of mechanical testing of composite specimens, before post cure, are shown in Table 3. Since the p-values for both ILSS and flexure tests are less than 0.05, a change in cure conditions has a significant effect on the mechanical properties of the composite laminate. Figure 8 depicts response surface plot of the effect of curing conditions on the ILSS of the composite panels.

The delamination resistance increases with a rise in curing temperature an improvement in degree of cure and crosslink density, with a rise in curing temperature and time , resulting in a better bond between the fiber and matrix. All samples failed by delamination between the central layers. ILSS is also shown to increase with a rise in the curing time.

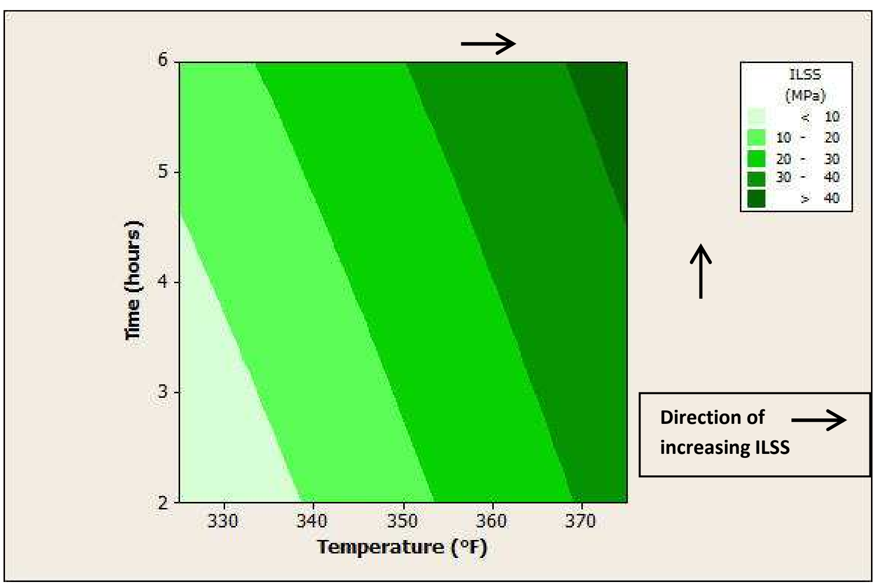

Figure 8. Contour Plot of ILSS vs Cure Temperature and Time

The laminate fabricated using a base line cure of 375 °F/ 6 hours exhibited the greatest ILSS of 41.06 MPa. A 7 °F drop in cure temperature is capable of producing a similar effect on delamination resistance, compared to a 1.5 hour drop in cure time. By interpolation, it is evident that a cure cycle of 360 °F for 6 hours can produce a composite laminate having an ILSS equivalent to one cured at the manufacturer recommended cure cycle of 375 °F/ 2 hours.

6.2. DROOP TEST

Droop due to thermal warping under a free standing post cure (400 °F for 2 hours) was studied and the maximum vertical displacement of the composite laminate was recorded using a dial gauge with a least count of 10^{-3} in. (Table 3). A very small amount of droop was observed in all laminates. The p-value, calculated from ANOVA, was found to be 0.612. Since p-value >0.05, a change in curing conditions, within the selected bounds of this study, showed no significant effects on the thermal warping of the composite panel. But, since the number of center point replicates is low, there is a possibility that the test conducted is not powerful enough to detect minute effects of changes in base cure cycle on droop.

6.3. VERIFICATION OF RESULTS

A composite panel was manufactured using a cure cycle of 360 °F/6 hours. The average cured ply thickness was 0.118 in. (2.99 mm), void content was 0.83% and the density was 1.56 g/cm^3. Five samples were removed and their ILSS was measured by short beam shear. The average ILSS was calculated to be 38.1 MPa. The samples failed by pure interlaminar shear, initiated by cracking of central layers, followed by delamination of the other layers (Figure 10).

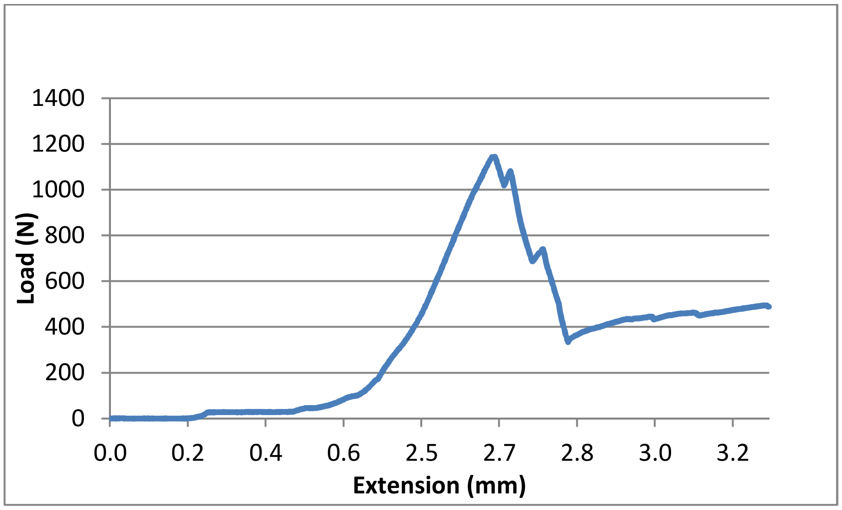

Figure 9. Short Beam Shear Test, Load vs Extension

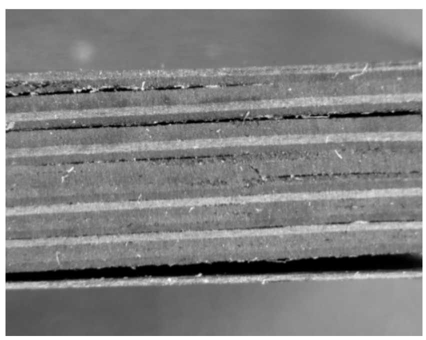

Figure 10. Cross Section of Tested Specimen

7. CONCLUSIONS

Bismaleimide composite laminates were manufactured successfully using the OOA process. Three different bleeders were evaluated for effectiveness, and one was selected for use in subsequent testing based on void contents measured by acid digestion. The selected bleeder was used to manufacture composite laminates for evaluation of cure cycle variation on ILSS and thermal warping after post cure. As expected, the mechanical properties increased with increase in cure temperature and duration. All cure cycle options were found to be capable of producing a panel with sufficient green strength to prevent warping during post cure. Based on the results of mechanical testing, a laminate cured at 360 °F for 6 hours can result in high interlaminar shear strength. Results of statistical analysis were verified. Future work in this area will investigate the effect of post cure cycles on performance of BMI composite laminates.

8. ACKNOWLEDGEMENTS

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II. Paper

MONITORING OF OUT-OF-AUTOCLAVE BMI COMPOSITES USING FIBER OPTIC SENSORS

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ABSTRACT

Bismaleimide (BMI) composites are used in applications that require good mechanical properties at high temperatures. In this paper, a Non-destructive inspection technique for BMI composites which can be used at high temperatures is presented. cavity based External Fabry-Perot Interferometer (EFPI) optical sensors have been developed and embedded in the laminates. These sensors are capable of operating in temperatures up to 800°C. The embedded sensors are used to perform real time cure monitoring of a BMI composite. The composite is cured using an out-of-autoclave (OOA) process. Once the composite is cured, the same sensors are used to measure mechanical performance of the laminate. The embedded fiber optic sensors were found to be capable of structural health monitoring as well as in-situ cure monitoring of an OOA cured carbon/BMI laminate.

1. INTRODUCTION

High performance Bismaleimide (BMI) composites are generally used in aerospace applications as they provide good mechanical performance at elevated temperatures. They are used as a high temperature substitute for toughened epoxy resins. A variety of processes can be used to manufacture composites like vacuum bag autoclave technique, hot press molding and Out-of-Autoclave (OOA) techniques. OOA methods can be used to manufacture high quality parts while providing great reductions in cost of manufacturing compared to traditional autoclave manufacturing. In the present work, Carbon fiber/ BMI composite laminates (IM7/AR4550 from Aldila Composites) are manufactured using the OOA process.

Mechanical properties of composites are strongly influenced by chemical and thermal events during cure. Stresses can be built up as a result of constrained thermal deformation during cure [1]. This can lead to microcracking of the matrix and thermal warping of the cured composite laminate. The residual stresses can reduce fatigue life of the manufactured composite [2]. Monitoring these effects in situ can help in optimization of cure cycles as well as damage detection in the composite laminate. Fiber optic sensors provide a cost effective means of evaluating residual stresses produced during the manufacturing process and detection of damage during cure [3-6].

Composites in aerospace applications can be exposed to prolonged and sometimes extreme service conditions. Optical fiber based sensors have many advantages like their compact size, resistance to corrosion, immunity from electromagnetic interference, and multiplexing capabilities. As a result of their compact size, embedding an optical sensor produces minimal distortion in the strain field within the specimen. Various optical sensors like Fiber Bragg gratings, Extrinsic Fabry-Perot Interferometer (EFPI) based sensors, Intrinsic Fabry-Perot Interferometer (IFPI) based sensors, Long Period Fiber gratings (LPFG), and combinations of these sensors have been used for health monitoring [7-9]. Feedback from structural health monitoring of composite materials can provide valuable information regarding in-service behavior of these materials.

EFPI based sensors are well suited for strain monitoring under elevated temperature conditions. Bragg gratings, and IFPI sensors can be used for strain monitoring [10,11] but they are very sensitive towards the ambient temperature. On the other hand, EFPI based optical fiber sensor is almost insensitive towards the ambient temperature when compared to its sensitivity towards the strain. Femtosecond (fs) laser fabrication yields thermally stable structures that can withstand high temperatures. An fs laser fabricated micro-cavity based embeddable assembly free EFPI sensor for strain monitoring at high temperatures is presented in this paper. The sensor is minimally sensitive to temperature. This sensor is embedded in Carbon fiber/ BMI composite laminated fabricated using an OOA process. Residual strain developed during cure is measured. The same sensor is then used to measure strain under tensile loading at room temperature as well as elevated temperatures.

2. METHODOLOGY

2.1. CURE MONITORING

Cavity based EFPI sensors were manufactured using a lab integrated femtosecond laser micromachining system at Missouri S&T. Out-of-autoclave cure of Carbon fiber/BMI composite laminates was performed and monitored using these EFPI sensors (Figure 1). Six layer unidirectional laminates were fabricated. First, three layers of BMI prepreg were placed on an aluminum mold. The fiber optic sensor was placed in the middle of the layup to avoid edge effects. A protective tube was used to protect the egress point which is prone to breaking. Three layers of prepreg were placed, followed by a layer of ETFE release film. A resin dam was placed around the perimeter of the laminate along with an edge bleeder. The layup was covered by a breather fabric and the sealed using a vacuum bag under full atmospheric pressure (29 mm Hg, 0.0038 MPa). The layup was placed in an oven. The fiber optic sensor was connected to an Optical Spectrum Analyzer (OSA) to monitor the cure. A 100 nm laser source (B&W TEK INC.) was used as input and a 3 dB coupler was used to send the signal to the sensor. The reflected signal was then recorded using an OSA.

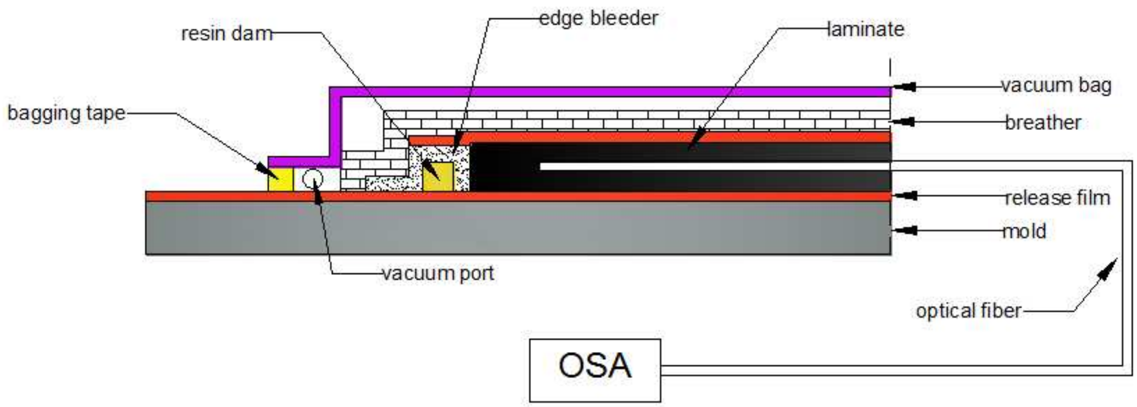

Figure 1. Cure monitoring layup, sectional view

Curing of the BMI takes place in two steps (Figure 2). First, the prepreg layup is heated to 121.1 °C (250 °F) for 1 hour. At this stage, the viscosity of the resin increases. Then, it is heated to 190.5 °C (375 °F) for two hours. The resin begins to cure as a rubbery viscoelastic material. Glass transition temperature (Tg) increases with a decrease in the fraction of unreacted monomer. As the Tg of the system approaches its cure temperature, vitrification phase begins [12]. On further curing, the Tg of the resin increases and the material transforms to a brittle glass-like structure. Volumetric contraction of the cured resin gives rise to compressive strains. The cured laminate is cooled to room temperature. As the cured laminate cools, residual stresses are induced due to a difference in the coefficient of thermal expansion between the fiber and the matrix. The compressive stresses increase further which corresponds to a shift in the wavelength in the optical fiber.

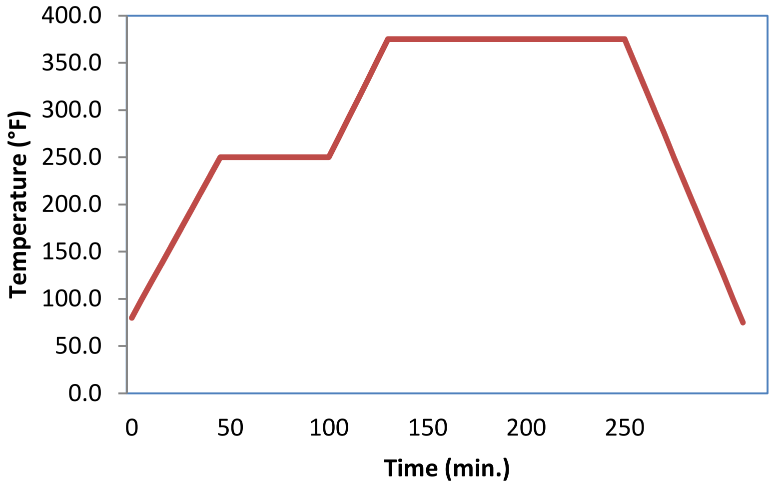

Figure 2. BMI prepreg cure cycle

2.2. MECHANICAL TESTING

Tensile testing samples measuring 304.8 mm x 25.4 mm (12 in. x 1 in.) were fabricated. Tabs, measuring 25.4 mm x 25.4 mm (1 in. square), were bonded at the ends using an epoxy adhesive. The strain response of the embedded sensors was investigated under tensile loading using an Instron 5584 testing machine and an OSA. Strain is transferred to the optical fiber by shear loads which lead to a corresponding change in wavelength of reflected light. Since cured BMI polymer is a brittle material, bonds between the sensor and resin can weaken and/or break under strain. This gives rise to hysteresis on the unloading curve. Strain transfer was studied under loading and unloading and unloading cycles to evaluate hysteresis, if any. The maximum strain limit of the sensor was evaluated. Mechanical tests were performed at room temperature. Samples were loaded to a maximum of 4000 µstrain which is the breaking point of the sensor at the cavity.

3. RESULTS AND DISCUSSION

3.1. CURE MONITORING

Figure 3 represents the cure monitoring of a $[0°]_6$ Carbon fiber/ Bismaleimide composite. The different phases associated with curing of the composite laminate can be discerned. In the first phase, the layup is heated to 121.1 °C (250 °F), in order to heat the resin and increase the mobility of reacting groups. At this point bonds between optical fiber and the resin are weak. The sensor shows a small shift in wavelength due to the temperature change. As the resin temperature increases to 190.5 °C (375 °F), the composite begins to cure. Bonding between the sensor and the matrix improves, and volumetric contraction of the resin induces compressive strain in the optical fiber which is shown as a corresponding decrease in wavelength.

As the degree of cure of the resin increases, it's Tg increases. At one point, the Tg of the system becomes equal to the curing temperature. This is the vitrification phase in the cure cycle. The material transforms from a viscoelastic material to a brittle glassy material, as the curing progresses. This is shown as a relatively flat region in the measured wavelength, followed by a change in the slope of the curve.. As the laminate cools to room temperature, the compressive strain increases due to thermal stresses. The residual thermal strain is calculated and the strain in the laminate was calculated to be 678 µstrain.

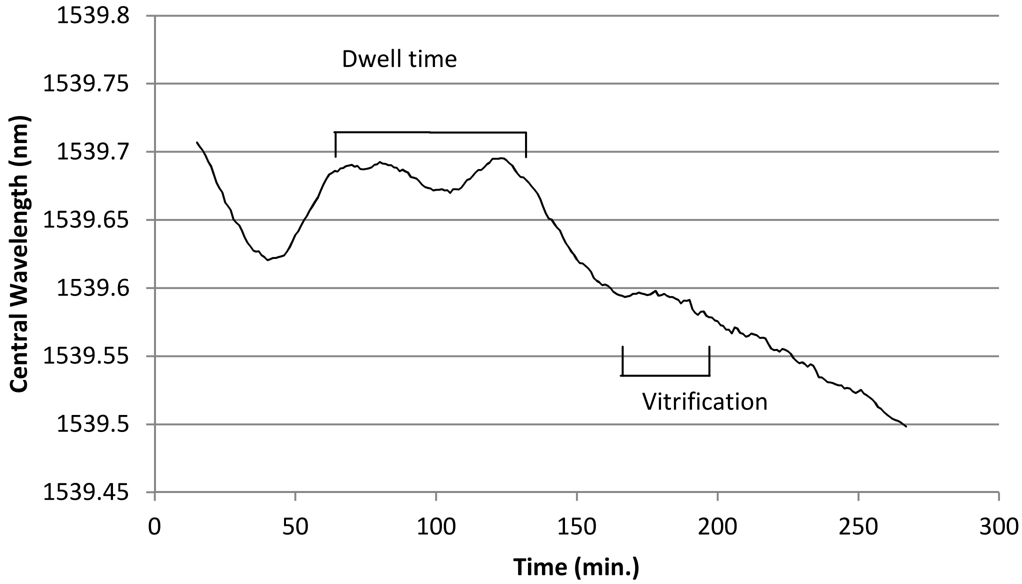

Figure 3. Cure monitoring of BMI composite laminate

3.2. MECHANICAL TESTING

Figure 4 shows the strain response of the sensor measured at room temperature. A crosshead speed corresponding to strain rate of 0.1 % per minute was used. The sensor response was recorded using an OSA. This was compared with the applied strain. The shift in wavelength was found to increase linearly with applied strain. A linear wavelength shift indicates existence of strong interfacial bonds between the optical fiber and surrounding resin. Slope of the loading curve was measured to be 36.22 °. This shows good correlation between measured strain and applied strain in the laminate. Little hysteresis was noticed when the sample was tested under room temperature. The unloading curve closely follows the loading curve. Interfacial bonds between the fiber and matrix were left intact when the laminate is loaded to a maximum of 4000 µstrain. A slope of 36° to 39° was recorded in successive experiments under the same conditions.

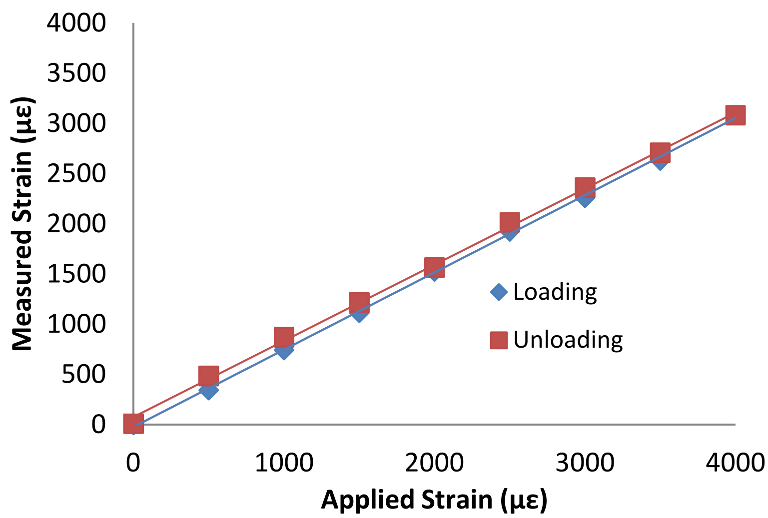

Figure 4. Tensile testing of specimens at room temperature

4. CONCLUSIONS

Cavity based fiber optic sensors were manufactured successfully using a femtosecond laser at Missouri S&T. These sensors were embedded into Carbon fiber/ BMI composite laminates manufactured using the OOA process. Cure monitoring was performed in-situ and the residual strain due to thermal expansion was measured. The residual strain during cure was measured to be 678 με. These sensors were then used to monitor strain in the sample under tensile loading. The sensor response was linear at room temperature. A strong interfacial bond exists between the fiber optic sensor and the composite laminate. Fiber optic sensors can be used in structural health monitoring of OOA cured BMI composite laminates.

5. ACKNOWLEDGEMENTS

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SECTION

2. CONCLUSIONS

Bismaleimide composite laminates were manufactured successfully using the OOA process. In the first part of the work, effect of processing variable on the quality of BMI composite laminates was studied. An effective bleeder was selected based on the results of void content tests on manufactured laminates. Effect of varying cure cycles on the green strength of laminates was investigated. All cure cycle options were found to be capable of producing a panel with sufficient green strength to prevent warping during post cure. Based on the results of mechanical testing, a laminate cured at 360 °F for 6 hours can result in high interlaminar shear strength. Results of statistical analysis were verified.

In the second part, cavity based fiber optic sensors were manufactured successfully using a femtosecond laser at Missouri S&T. These sensors were embedded into Carbon fiber/ BMI composite laminates manufactured using the OOA process. Cure monitoring was performed in-situ and the residual strain due to thermal expansion was measured. The residual strain during cure was measured to be 678 με. These sensors were then used to monitor strain in the sample under tensile loading. The sensor response was found to be linear at room temperature. These sensors are capable of performing structural health monitoring.

APPENDIX

A.1 Effect of base cure cycles on flexural strength of OOA cured BMI laminates

Flexural tests were performed according to ASTM D790 at a span to thickness ratio of 40:1. The samples were loaded using a three point bending fixture (figure 3.1) at a rate of 0.4 in. per min on a loading span of 6 in. Flexure stress was calculated using equation below.

$$\sigma = \frac{3PL}{2bd^2} \tag{3.1}$$

Where, P =load in N, L=span length, b=sample width, d=depth of the specimen

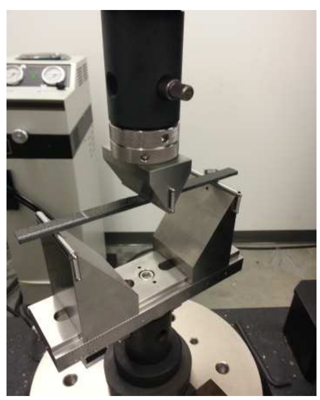

Figure 3.1. Flexure Test Setup

Figure 3.2 shows a contour plot of the change in flexure stress at failure of panels cured at varying cure cycles. When loaded under large span to depth ratios (40:1), the failure of test specimens is dominated by flexure. In composites that have low compressive strength compared to tensile strength, failure is initiated by compressive fracture of upper layers followed by delamination.

In case of samples cured at 325 °F, failure was dominated by delamination due to low ILSS. However, in case of laminates cured at temperatures of 350 °F and 375 °F, flexural failure was observed. The contour plot shows an increase in flexure strength with an increase in cure temperature as well as cure time. Flexure strength of 431.91 MPa was obtained using a base line cure of 375 °F/ 6 hours. Reducing the cure time to 2 hours at 375 °F can result in flexure strength of 400 MPa. This can also be obtained using a cure cycle of 354 °F and 6 hours. In order to fabricate a laminate that has high ILSS as well as flexure strength, a cure cycle of 375 °F/ 4.5 hours can be used.

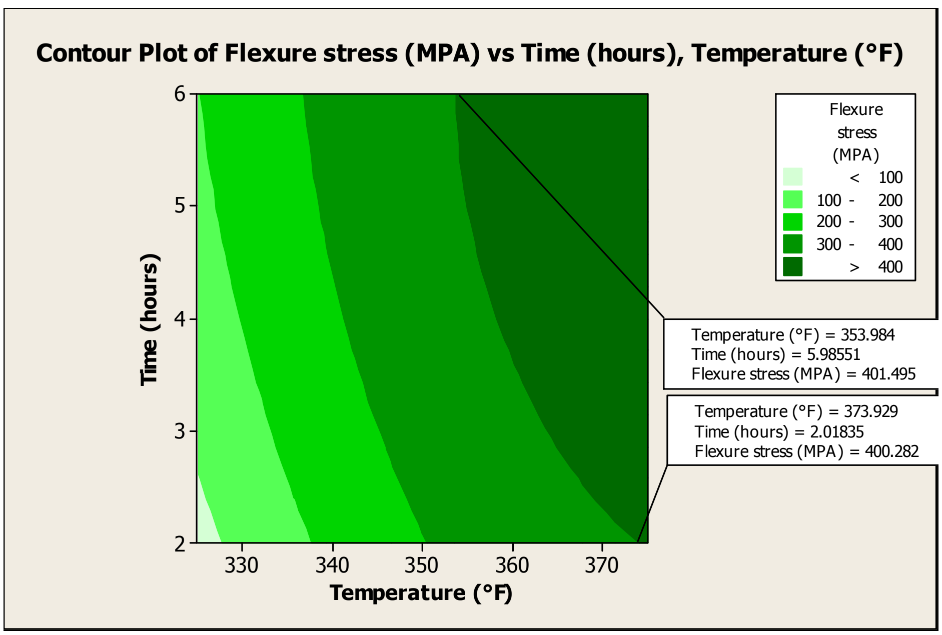

Figure 3.2 Contour plot of variation of flexure strength with cure time and temperature

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