Summer 2019

Performance evaluation of BMI resin system for thin-ply composites

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Recommended Citation

Rangapuram, Manoj Kumar Reddy, "Performance evaluation of BMI resin system for thin-ply composites" (2019). *Masters Theses*. 7948.
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PERFORMANCE EVALUATION OF BMI RESIN SYSTEM FOR THIN-PLY COMPOSITES

by

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A THESIS

Presented to the Faculty of the Graduate School of the

MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE

in

MECHANICAL ENGINEERING

2019

Approved by:

K. Chandrashekhara, Advisor
L. Chen
L.R. Dharani

PUBLICATION THESIS OPTION

This thesis consists of the following one article, formatted in the style used by the Missouri University of Science and Technology:

Paper I: Performance Evaluation of BMI resin system for Thin-ply Composites (Pages 5-23) has been submitted to Proccedings of the Composites and Advanced Materials Expo (CAMX) Conference, Anaheim, CA, September 23-26, 2019.

ABSTRACT

Composites materials are increasingly being used in aerospace applications over the past few years. The unique properties like high strength to weight ratio, thermal stability, fatigue and corrosion resistance set them apart from the conventional materials. Composite materials are well suited for the applications where weight is the primary concern in the design. Composites structures are vulnerable to mechanical as well as thermal loadings. Transverse micro-cracking and delamination are the most common type of failures in composite materials. The thickness of the ply being used play a key role dictating the properties of the resultant composite structure. As the ply gets thinner the properties get better. Thick laminates are more susceptible to micro-cracking than thin laminates. Thereby, to manufacture laminates resistant to micro-cracking and delamination it is advised to use thinner plies. In this work, a BMI hardened prepreg system was used to prepare the laminated composites. Thin and thick ply laminates were used to make the composite panels .Mechanical testing was performed on the panels to evaluate the performance of thin-ply and thick-ply laminate system

ACKNOWLEDGMENTS

I would like to express my deepest gratitude to my academic advisor Dr. K Chandrashekhara whose guidance and advice have been invaluable throughout my master's degree. His constructive suggestions have been immensely useful during the course of my graduate research.

I thank the members of my advisory committee, Dr.Liyani Chen and Dr.Lokeswarappa Dharani for their valuable time and advice in the review of this dissertation.

I am indebted to the members of the Composite Research group, especially Mr. Dasari Sivasai Krishna and Mr. Robert Meinders, who worked with me extensively during my research and this study would have been impossible without their help.

I must also thank our industry collaborators, Dr. Nagaraja Iyyer, Dr. Mehdi Naderi (TDA) and Mr. Robert Stratton (Stratton Composites) for their valuable input.

Finally, I also express my gratitude to my family and friends for all the love and encouragement.

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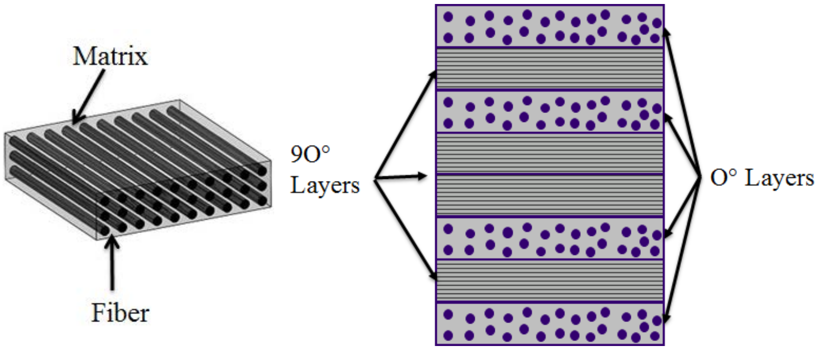
1. INTRODUCTION

Composites are defined as structures made from two or more components to form a new material system with enhanced properties. The current work focuses on fiber reinforced polymer composite. The fibers are made of carbon while the matrix material comprises of thermoset polymers such as epoxy or Bismaleimide (Figure 1.1). High temperature systems such as BMI based composites are widely used in high temperature aerospace applications due to their superior properties compared to epoxy resins. They also offer better mechanical properties like tension, bending, superior chemical and corrosion resistance [1].



(a) Composite lamina (b) Cross-ply composite laminate

Figure 1.1. Composite lamina

Composite structures have been widely utilized in diverse applications ranging from housing, automobile to aerospace industries due to their high strength-to-weight ratio and design flexibility. A composite laminate consists of a stack of multiple layers or “lamina”. Figure 1.1 (b) shows a cross-ply composite laminate where subsequent layers have fibers oriented perpendicular to each other. The orientation of the plies can be tailored to obtain a specific combination of properties, according to design requirements.

1.1. PREPREG LAMINATES

The raw material that is used to manufacture a composite laminate is called a “prepreg”, or pre-impregnated fibers. The prepregs maybe unidirectional or woven infused with uncured resin. The prepreg tapes are cut into required dimensions and are sequentially laid to result in the desired properties. Similar layup schemes like unidirectional, cross-ply, angular orientations are used to tailor the required properties in the resulting composite. In this study unidirectional prepreg tapes with two different aerial weights 30-40 gsm and 150 gsm were manufactured using Toray T300 3k and 12k carbon fibers and toughened BMI resin system.

1.2. QUASI-ISOTROPIC LAYUP-SCHEME

“Isotropic” means having same properties in all the directions. “Quasi-isotropic” means having same properties in-plane direction. This can be achieved by randomly orienting fibers in all the directions or having fibers oriented in equal ratios in all the directions. Simple quasi-isotropic layups are achieved by 45^o, -45^o, 0^o, 90^o orientations whereas 0^o, 60^o and 120^o can also be used to generate the layups. In this study $[45^o/-45^o/0^o/90^o]_{6s}$ was used to manufacture both thin and thick-ply laminated composites.

1.3. CURING

As the laminates are toughened with a resin system these resins have to be cured to cross-link and result in the superior properties. Every resin system has a unique “cure cycle”. Cure cycle is defined as the control of time-temperature-pressure of a thermosetting resin system to change its state from liquid to solid [2].

1.4. MANUFACTURING

Aerospace composite materials are generally manufactured in an autoclave under high temperature and high pressure. The combination of high temperature and pressure ensures low void content and good part consolidation. The other manufacturing process used is Out-of Autoclave (OOA) process which is relatively new and low-cost replacement to autoclave process [3,4]. The OOA process requires less investment and also enables us with design flexibility in manufacturing the components. The size of the autoclave no longer dictates the part size [5]. During OOA processing, sandwich components can be co-cured, a process where adhesive and the prepreg is cured simultaneously, resulting in further time and cost reduction. Manufacturing co-cured sandwich panels, out-of-autoclave, eliminates core crushing which can occur at autoclave pressures [6] and enables the use of lighter cores.

The pressure inside the autoclaves are so high that the voids are suppressed. But in the OOA as the pressures are relatively low the manufactured parts may be prone to higher void content. The vacuum-sealing in the OOA process should be done with utmost care to avoid entrapment of gases. The areas of porosity may vary from plies, tows or individual fibers. During the course of curing, as the melting point is reaches the resin fills the gaps in this pores resulting in a void-free structure [7]. Studies have been performed to establish a relationship between the porosity and mechanical properties of the manufactured part. [8]. In OOA process venting of the entrapped air is the key to manufacture void-free parts. Therefore, the processing parameters for OOA processing of composite laminates and sandwich structures should be carefully established, in order to reap the benefits of low manufacturing costs. Therefore, the current work involves evaluation of OOA cured composite laminates and sandwich structures and optimization of process parameters to reduce void contents.

PAPER

I. PERFORMANCE EVALUATION OF BMI RESIN SYSTEM FOR THIN-PLY COMPOSITES

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ABSTRACT

Thin ply composites are gaining a lot of attention in composite industry because of the vast design flexibilities they offer. Thin-ply composites reduce the weight of the component and also offer superior mechanical properties. Ply thickness plays a prominent role in controlling the mechanical properties of composites. The thinner the ply the better the properties. The properties are improved because the thinner ply results in lower residual stresses after curing at elevated temperatures due to coefficient of thermal expansion of composites. Thicker laminates are more vulnerable to micro-cracking than thin laminates. High temperature systems such as bismaleimide based composites are being used in the industry due to their better characteristics compared to conventional epoxy systems. 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 as well as corrosion and chemical resistance. In this study, a novel high temperature out-of-autoclave BMI resin (BMI-1OOA) was used to prepare the thin prepregs. The resin system was evaluated to obtain the neat resin properties and apply it to the prepreg system. Mechanical testing of the resin system was performed using tension test following ASTM standards. Differential scanning calorimetry and thermo gravimetric analysis were also performed to evaluate the glass transition temperature and thermal properties of the resin system. Toray T300 3k and 12k carbon fibers with 30-40 gsm and 150 gsm aerial weight respectively were used to produce thin prepregs. The mechanical properties of the thin prepregs were evaluated by testing composites in Impact. The thin-ply composites performed better than thick-ply composites under impact loading.

1. INTRODUCTION

Thin-ply laminate systems are gaining the interest of the industry in the recent years. A significant amount of progress has been made in the development of thin-ply composites. The main aim of this technology is not only to produce thinner and lighter laminate structures but also provide improved strength and damage resistance due to innovation in laminate design space and positive size effects [1]. The striking advantage of employing thinner plies in a given structure at a constant predetermined thickness is the ability to use an increased number of ply orientations to achieve the optimal solution within the existing design space. This fact is particularly important for the existing thin laminates for which only two or three orientations can be selected to meet the classical design constraints like prepreg thickness, laminate symmetry and fraction of fibers at 90° orientation. Replacing a 300 gsm standard prepreg with a thinner prepreg such as a 30 gsm will allow the designer to propose more optimal lamination schemes such as [0°/45°/90°/-45°] or even more complex schemes instead of a basic [0°/60°/-60°] layup.

The other prime benefits of using thin plies instead of thick plies is suppression of sub critical damage such as micro cracking and delamination as well as an extraordinary improvement to the fatigue life and increased damage tolerance [2-6]. For the development of novel materials (thin-ply), a thorough understanding of the advantages and drawbacks of the system from material properties, processing, manufacturing, design, and cost-effectiveness is quite necessary. Ply thinning technologies have been developed in recent times to fabricate thin plies. One such technology is spread tow thin-ply technology in which the tows are continuously opened and spread to produce flat and straight plies with thickness as low as 20 µm.

Traditionally, the two main components that make the composites are fibers and reinforcement. The fibers are usually made from strong and stiff material providing strength and stiffness to the composite structure. Moreover, fibers alone cannot provide the above-mentioned properties owing to their size. Reinforcements or matrix materials generally have low normal strength (tensile and compression) but they provide with the following functionalities: aligning of fibers, transfer of loads between fibers and adjacent layers of plies, and also protecting the fibers from environmental effects [7]. Matrix material used in this research is a novel Bismaleimide (BMI) resin system for OOA process. BMIs are a comparatively newer class of the thermosetting polymers. They are gaining importance because of their distinct features like the retention of physical properties at elevated temperatures and damp environments as well as sustained electric properties over a broad temperature range. The steady optimization of thermal, mechanical, and electrical properties make them a viable source in electronics and composites. They possess high strength and excellent long-term creep resistance. They are notably used in high performance fiber reinforced polymers as matrix material. Typically, they require curing temperatures of 150 °C or more and higher post cure for high temperature applications such as racecars and military grade airplanes.

The first studies on evaluating the mechanical characterization of thin-ply composites was done by Tsai et al. [2]. In their work, they tested quasi-isotropic composite panels made from carbon-epoxy. Tsai tested both thin and thick plies using unnotched tensile tests in static and fatigue loading, open hole tensile test on the quasi-isotopic panels, and compression after impact. In the unnotched tests, the thin-ply laminate exhibited higher ultimate strength than the thicker laminates. A significant amount of damage was visible on the thicker laminate before failure while the thinner laminate exhibited linearly elastic behavior until failure. Fatigue life of the thin plies was also quite impressive. After 50,000 fatigue cycles there was no change in the strength of the thinner ply whereas there was significant drop in the strength of the thicker laminate. Open-hole compression test followed a similar trend. There was limited damage around the hole in the thinner ply while progressive delamination and transverse micro-cracking was observed in the thick ply laminate. Impact studies on the quasi-isotropic panel suggested there was reduced delamination area in thin-ply compared to the thicker ply. Several other studies also proved that the thin ply laminated composites improve the mechanical properties by delaying delamination and transverse micro-cracking [8-10]. Multiple studies conducted on thin-ply laminated composites proved that they can delay and even suppress transverse micro-cracking and edge delamination in various loading conditions such as static, fatigue, and impact [11-13]. The composite manufacturing process has several factors affecting it such as fiber alignment, waviness, residual strains, porosity, and resin rich zones which introduces another factor related to the size effect that controls the performance of the composite.

From the existing literature on size effects and delamination the thin ply composites perform better than any traditional composite material. But there is very limited experimental data and models that support the statement. Most of the research to date has dealt with unidirectional thin ply prepreg laminate system [2,3,10,14]. From this discussion it is quite evident that further analysis and testing of thin-ply laminated composites is necessary.

The present work is divided into two parts. The first part deals with the BMI neat resin characterization and properties evaluation. Neat resin cure kinetics will be studied using thermogravimetric analysis (TGA) and digital scanning calorimetry (DSC). Neat resin mechanical properties will be evaluated using tension. The latter part deals with the manufacturing of Carbon/BMI composite panels and experimental evaluation.

2. EXPERIMENTATION

2.1. MATERIALS

In the present work, unidirectional prepreg tapes with two different aerial weights 30-40 gsm and 150 gsm were manufactured using Toray T300 3k and 12k carbon fibers and toughened BMI resin system. The resin accounts for 35% by weight in the prepreg system. The advantage of this resin system is it has low tack and drape compared to conventional epoxy system and has a shelf life of about two weeks. Raw materials like vacuum bag, breather, release film, bagging tape, and edge bleeders are used in a conventional out of autoclave (OOA) process. The cure cycle followed is shown in Figure 1 below. The temperature is first raised to 143 °C (290 °F) at a rate of 4 °C/min and held for one hour before the base cure of 190 °C (375 °F) for two hours to mobilize the reactive groups.

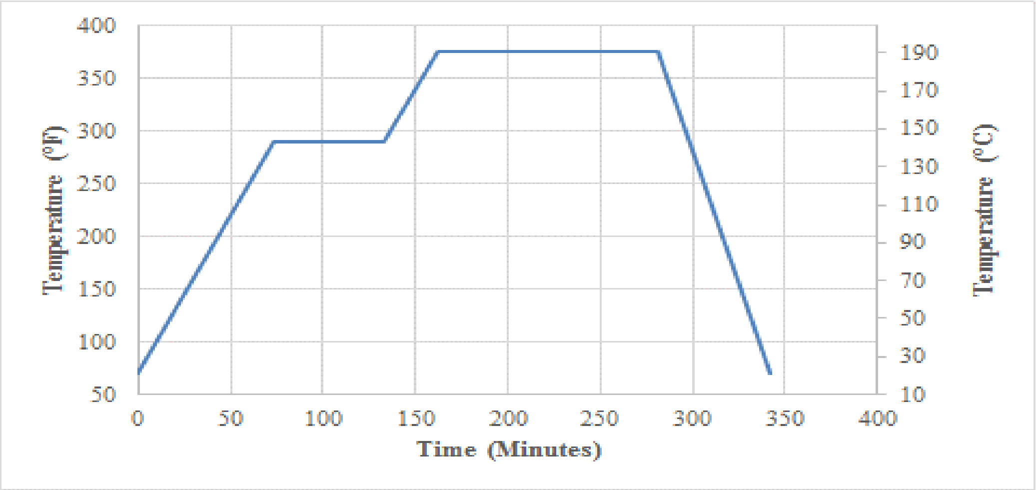


Figure 1. Cure cycle of BMI-OOA resin

The resin system used in impregnation of the prepregs is a novel high temperature BMI resin (BMI-1OOA) manufactured by Stratton Composites. The neat resin samples were fabricated to evaluate the mechanical properties.

2.2. METHODOLOGIES

2.2.1. Neat Resin Panels Fabrication. Neat resin panels were fabricated using BMI resin system. The neat resin was poured into molds and then degassed to remove absorbed air during the handling and finally cured. Neat resin tension coupons were fabricated following standard ASTM coupon dimensions.

2.2.2. Composite Laminate Manufacturing using OOA Process. The composite laminates were manufactured using OOA prepreg process. To study the difference between thin and thick laminates a quasi-isotropic layup scheme $[45^o/-45^o/0^o/90^o]_{6s}$ was used to fabricate composite panels.

Initially, the aluminum baseplate was cleaned and coated with Ethylene Tetraflouroethylene (ETFE) release film. The unidirectional prepreg tapes were cut to required size and placed according to the specified lamination scheme. A teflon coated edge bleeder was used to provide a passage for the entrapped air during the process. The entire setup was sealed using vacuum bag. The vacuum pressure was 28 in. Hg. Debulking was done for every four layers for 5 minutes to remove the entrapped air from the laminate stack. The bagging scheme followed in the process is shown in the Figure 2. The laminate layup was then sealed using double bagging and cured in an oven. The prescribed base cure was 190 °C (375 °F) for two hours and post cure of 210 ºC (410 °F) for 4-6 hours.

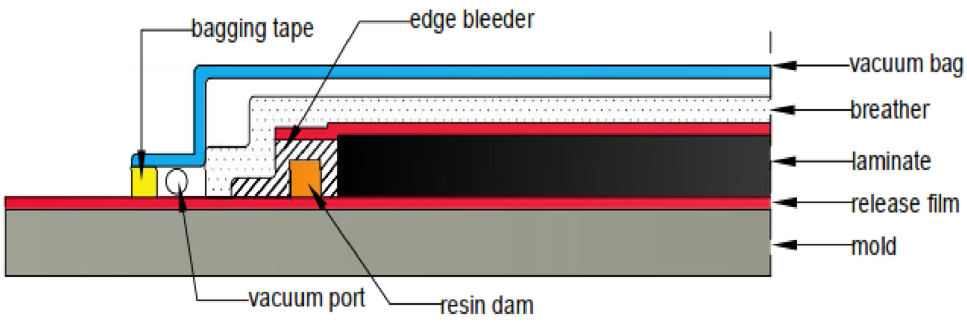


Figure 2. Layup scheme used in OOA process [15]

2.3. MATERIAL CHARACTERIZATION

2.3.1. Cure Kinetics of Neat Resin.

2.3.1.1. Thermogravimetric Analysis (TGA). TGA is a thermal analysis that determines the thermal degradation of a material. In this paper, thermal degradation of the cured and uncured neat resin was performed on TA Instruments Q50 Thermogravimetric Analyzer. Sample sizes of 5-10 mg were taken in a platinum pan and analyzed. In TGA, the weight change of the resin was measured as a function of temperature to understand the thermal degradation of the resin. Samples were heated from room temperature to 800 ^{o}C at 10^{o}C/min standard heating rate in air and the degradation pattern was recorded.

2.3.1.2. Differential Scanning Calorimetry (DSC). DSC is a thermo-chemical analysis used extensively to study the thermal properties of a resin. DSC is generally used to study the cure kinetics of the thermosetting resins. In this work a TA Instruments Q2000 DSC was used to analyze both cured and uncured neat BMI-1OOA resin to understand the cure behavior of the resin. Samples ranging from 5-10 mg were encapsulated in aluminum pans and inserted into the machine. Dynamic heating rate of 3 °C/min with amplitude of 1 °C was used to determine the glass transition temperature.

2.3.2. Neat Resin Tension Test. Tension test of neat resin was performed on Instron-5985 test frame according to ASTM-D638. BMI-1OOA neat resin samples were fabricated at Missouri S&T. Neat resin samples were fabricated using standard aluminum mold following the prescribed cure cycle. Neat resin coupons are shown in Figure 3. Neat resin tensile test was performed to evaluate the modulus and the ultimate tensile strength of the resin system.

Figure 3. Neat resin tension speicmens

2.3.3. Laminate Impact Test. A quasi-isotropic $[45^o/-45^o/0^o/90^o]_{6s}$ layup scheme was used to manufacture composite panels through OOA process. Impact test was performed on thick and thin panels to evaluate the difference between the damage evolution and delamination between the panels. Impact test was performed using a Dynatup Instron Model 9250 impact testing machine to conduct the low-velocity impact tests. In this test, the effects of two energy levels 10 J and 15 J were evaluated on the deflection, energy and overall damage.

(a) Thin-ply composite at 10J and 15J (b) Thick ply composite at 10J and 15J

Figure 4. The impacted composite panels

3. RESULTS AND DISCUSSION

3.1. CURE KINETICS STUDY

Thermogravimetric Analysis (TGA) was performed on uncured resin used for preparing the neat resin samples. The sample was heated at 8 °C/min in air. Minor weight changes were observed above 160 °C and final degradation occurred with an onset point of 410 °C. The complete burn-off of resin occurred around 640 °C. The insight of this change in mass of the resin with change in temperature was necessary to perform Digital Scanning calorimetry (DSC). This test basically provides us with the information of the temperature range through which our resin can be used without any mass loss and without any emissions. It gives us the insight in which particular temperature range DSC can be performed. Both these tests are done as a part of cure kitenics study of any thermosetting resin.

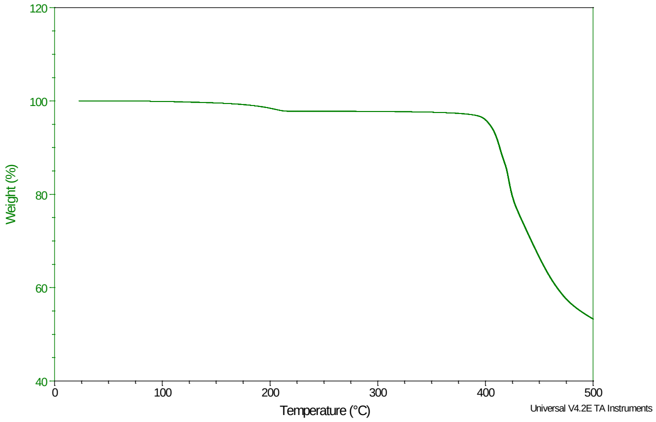


Figure 5.TGA curve of uncured BMI resin in air

Modulated Differential Scanning Calorimetry (MDSC) was performed on samples of neat resin. Specimens were subjected to MDSC from 30 °C to 380 °C at 3 °C/min with amplitude of 1 °C. From the MDSC there appear to be two distinct T_g at 235 °C and 310 °C for the BMI specimen. This seems to indicate an incomplete heat treatment or post cure of the resin. The DSC results suggested additional post-cure was required for the DSC samples that were manufactured.

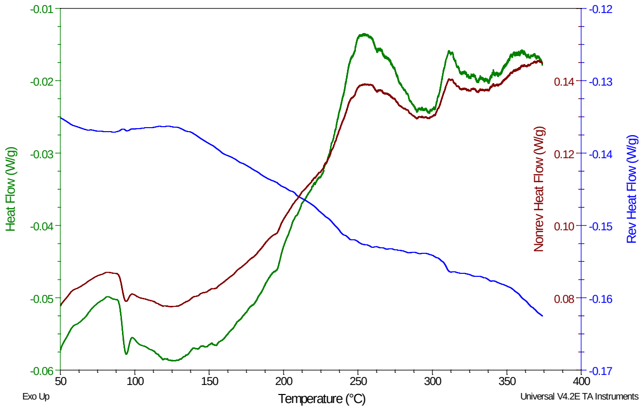


Figure 6. MDSC plot of heat flow through BMI resin sample

3.2. NEAT RESIN TENSION TEST

Five specimens were tested using an Instron-5985 test frame. All the samples showed brittle behavior and there was no plastic region in the stress-strain curve (Figure 7). As it can be seen from the graph that there is no yielding region and material fails dramatically. Thus, ultimate strength is calculated based upon the maximum stress the material withstood before failure. Calculated tensile modulus and ultimate tensile strength are shown in Table 1. All the samples failed within the gauge section.

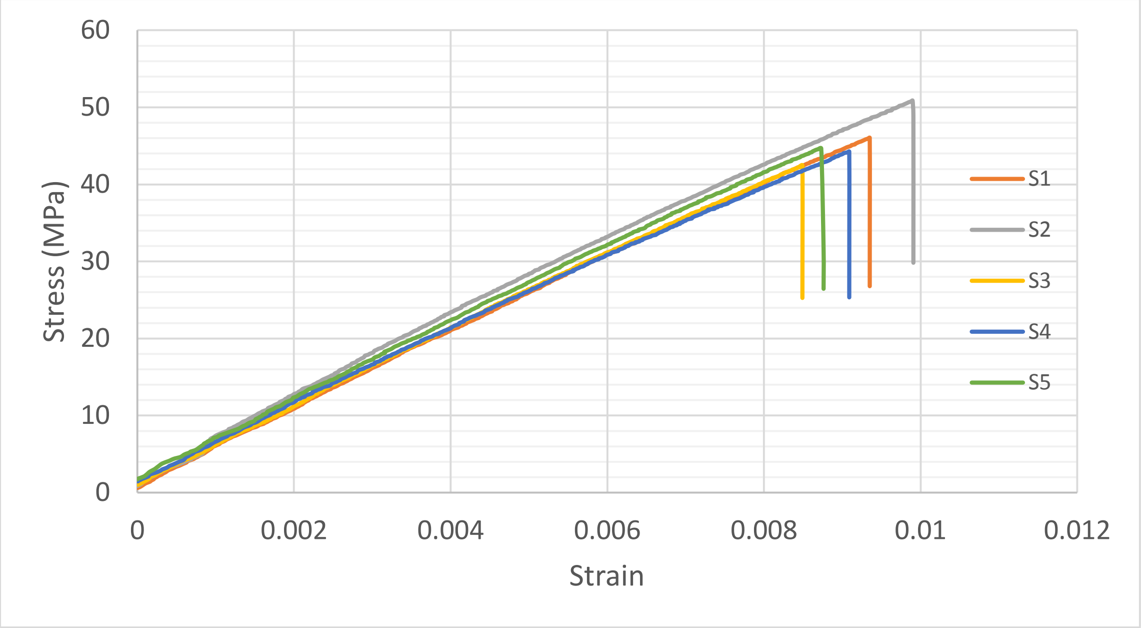


Figure 7. Stress-strain curve from neat resin tensile testing

Proper validation methodologies were followed for the specimens according to the respective ASTM standards. All the results were analyzed and plotted using Microsoft Excel. Only straight regions of the stress-strain curve were considered for modulus calculations.

Table 1. Results from neat resin tensile testing

Specimen	Ultimate strength (MPa)	Young's modulus (MPa)
1	46.06	5023.92
2	50.85	5494.46
3	42.47	5090.98
4	44.24	4956.37
5	44.65	5054.18
Average	45.65	5123.98
Standard deviation	3.17	212.90

3.3. IMPACT TEST

The results of the low velocity impact tests on thin and thick panels are presented in Table 2. Figure 9 refers to the energy variation with time during the test. The initial spike indicates the transfer of energy from the impactor to the panel. The latter part of the curve indicates the reaction force exerted by the plate on the impactor. Figure 10 depicts the load vs time at two different energy levels 10 J and 15 J. The peak load was shifting to the left with an increase in the impact energy in both thin and thick laminate panels. Figure 11 shows the velocity vs time graph during the impact test. Figure 12 indicates the load vs deflection. The ascending portion indicates the bending history of the specimen under impact loading and the descending portion indicates the rebounding of the impactor and softening of the composite. The load-deflection curve will be closed as long as the long as the impactor does not penetrate through composite. The area under the curve gives the absorbed energy. As it can be seen, the effect of load on deflection is increased with increasing energy. The impact damaged region of 15 J is greater than 10 J in general. But the impact regions on thick and thin panels at same energy levels were compared to evaluate the nature of the ply.

Table 2. Results from impact test

Type	Energy level	Peak load (kN)	Deflection at peak load (mm)	Energy to max load (J)	Impact velocity (m/s)
Thin	10J	11.8495	0.9263	5.8648	1.8028
Thin	15J	12.3106	1.2865	5.4724	2.2049
Thick	10J	11.6075	1.081	5.4727	1.8039
Thick	15J	13.2858	0.9565	6.1469	2.2078

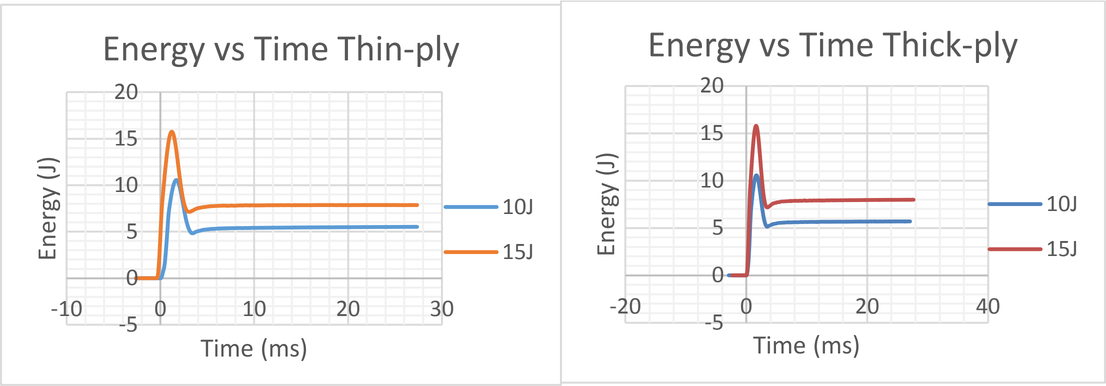
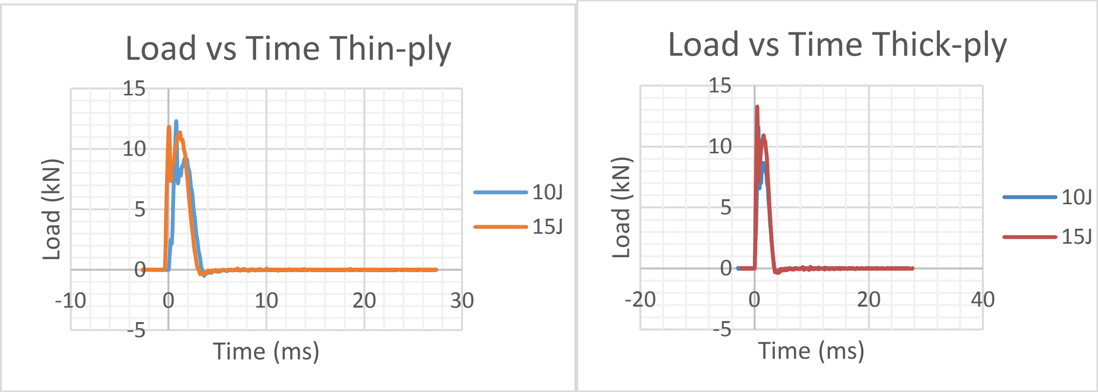
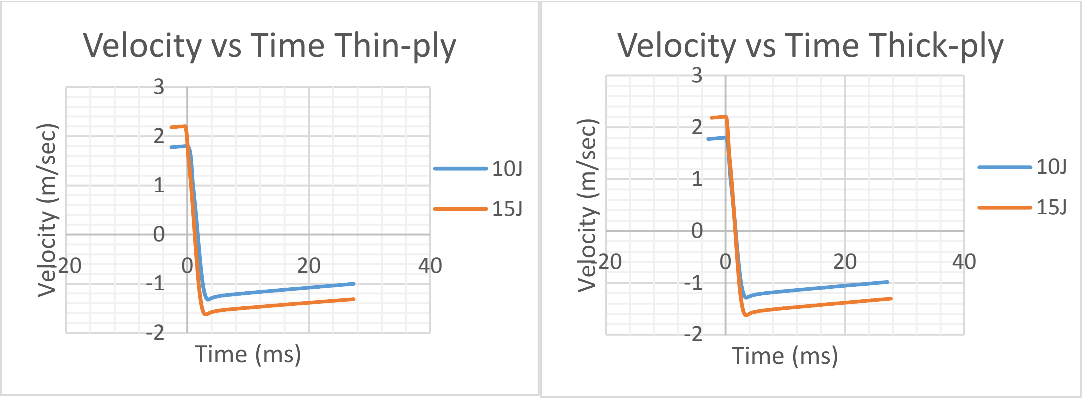


Figure 8. Energy vs time in low-velocity impact tests at 10J and 15J


Load vs Time Thin-ply

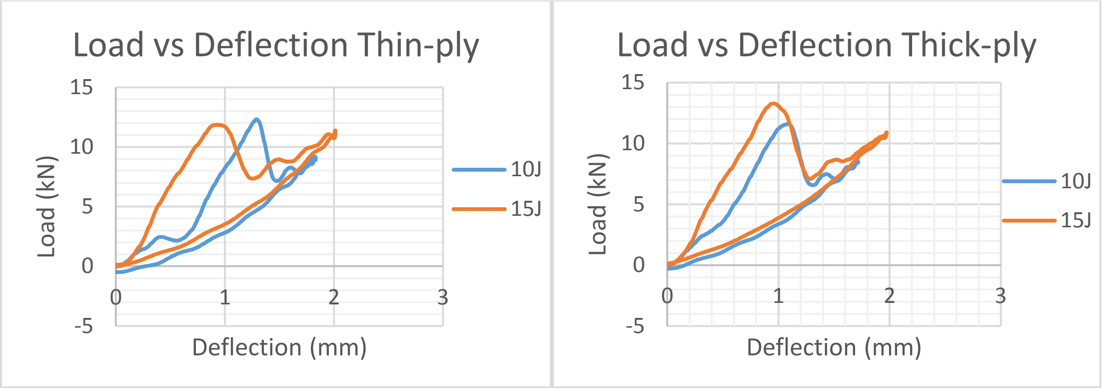
Load vs Time Thick-ply

Figure 9. Load vs time in low-velocity impact tests at 10J and 15J


Velocity vs Time Thin-ply

Velocity vs Time Thick-ply

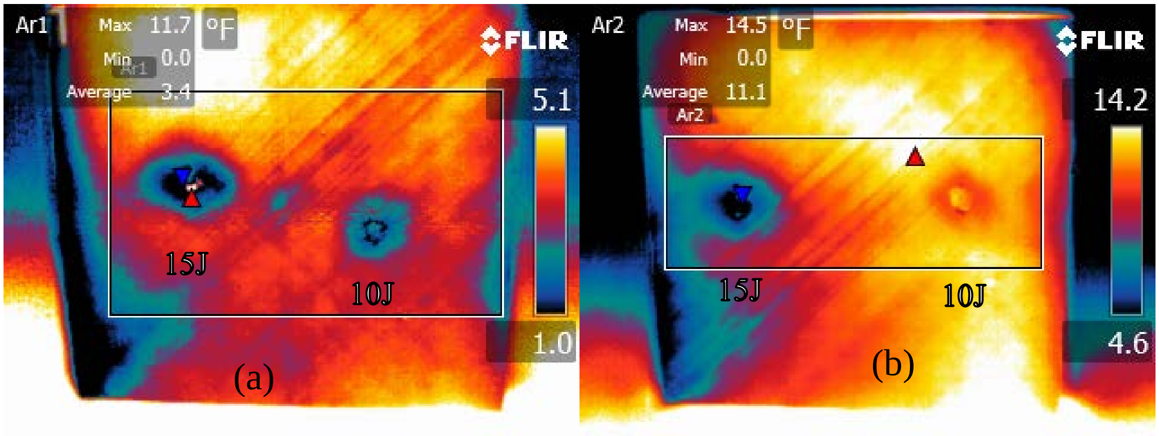
Figure 10. Velocity vs time in low-velocity impact tests at 10J and 15J


Load vs Deflection Thin-ply

Load vs Deflection Thick-ply

Figure 11. Load vs deflection in low-velocity impact tests at 10J and 15J

The comparison plots of thin-ply and thick-ply laminated composites (Figures 9-12) show almost similar information with minor differences. This indicates the performance of thin-ply system is almost equal to the thick-ply laminated system. The failure patterns are further observed using NDI technique and further understanding of the characteristics is done in the following section. Infrared-Thermography is a nondestructive investigation (NDI) technique generally employed to analyze a part or a component based on the heating and cooling patterns. Infrared (IR) thermography was performed on the impacted specimens to evaluate the impact damage on both thin and thick composite panels. Figure 13 show the IR bitmap image of the impacted specimens.



(a) Thin-ply composite panel at 10J and 15J (b) Thick-ply composite at 10J and 15J

Figure 12. IR-bitmap images of impacted specimens

The IR-bitmap images were analyzed in an image processing software called Image J and the impact area was measured. The measured impacted area for thin and thick panels at two energy levels 10 J and 15 J is tabulated and shown in the table below.

Table 3. Quantified impacted areas in Image J software

Energy level	Area (mm^2)	
	Thin-ply	Thick-ply
10	30.213	34.455
15	45.594	48.249

From the observed IR-bitmap images and the quantified areas of the impacted region it is evident that the thin-ply composite panel had less damage at both 10 J and 15 J energy levels compared to the thick-ply composite.

4. CONCLUSIONS

The novel BMI resin system (BMI-1OOA) was successfully cast into tension molds and mechanically characterized. Cure kinetics evaluation of the resin system was performed using TGA and MDSC studies. Unidirectional prepreg tapes with two different aerial weights 30-40 gsm and 150 gsm were manufactured using Toray T300 3k and 12k carbon fibers. A quasi-isotropic $[45^o/-45^o/0^o/90^o]_{6s}$ layup scheme was followed to manufacture both thin-ply and thick-ply laminate composite panels. The manufactured panels were subjected to low-velocity impact testing at 10 J and 15 J energy levels. Thermography studies performed on the impacted panels suggested that the impact damage was comparatively less on the thin-ply laminated composite than thick-ply at same energy level. This was further quantified by image processing the thermal bitmaps of the impacted specimens indicating the impact areas were smaller on the thin-ply laminate composite than the thick-ply laminate composite.

ACKNOWLEDGEMENT

This work is funded by Technical Data Analysis Inc.,(TDA), Falls Church, VA 22042.

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SECTION

2. CONCLUSIONS AND RECOMMENDATIONS

2.1. CONCLUSIONS

The paper used in this thesis primarily deals with the neat resin characterization. The neat resin properties are obtained to understand the performance of the resin used in the prepreg material. Essentially, the main concentrated factor being the differentiation between the performance of thin-ply and thick-ply laminate system. Different ply thickness have been used in manufacturing of both thin and thick-ply system. The performance of the laminates were tested under low-velocity impact tests. Both delamination and fiber fracture were observed in thin and thick-ply laminate system. But, the impacted area of delamination was comparatively less in thin-ply laminated composite compared to then of thick-ply laminated composite at same energy of impact. IR-thermography was performed to further validate the results.

2.2. RECOMMENDATIONS

The research presented here can be extended in many ways. The experimental work in this paper deal with manufacturing of composite panels with quasi-isotropic layup scheme for impact characterization. A different layup scheme can be used and the comparative study on the performance of the manufactured composite can be done. Different tests like tension, compression, open-hole compression and compression after impact can be done differentiate the properties of thin-ply and thick-ply laminate system.

Samples at various stages of failure can be obtained during mechanical testing and studied using Scanning Electron Microscopy (SEM) to study micro-cracking and delamination in thin-ply composites.

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VITA

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