Thermal and Mechanical Characterization of C/C Composite Produced via Highly Processable BODA-Derived Precursor Resin System

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Carbon/Carbon (C/C) composites are critical materials for high-temperature applications, but their manufacturing processes remain constrained by decades-old techniques. Bis-ortho-diynylarene (BODA)-derived resins offer a promising solution by enabling faster processing through innovative chemical mechanics. This report outlines the ongoing fabrication of a BODA-derived resin (BDR) C/C composite using compressive molding. Preliminary progress includes the development of fabrication techniques, with plans for future mechanical characterization through tensile testing and thermal cycling to assess the material's performance.

I. Nomenclature

$BODA$	=	Bis-*ortho*-Diynylarene
BDR	=	BODA-Derived Resin
C/C	=	Carbon/Carbon

II. Introduction

Carbon/carbon (C/C) composites, consisting of carbon fiber tows and a carbonaceous matrix, are a class of extreme materials known for their extreme strength and extremely high thermal stability. They are widely used in high temperature applications in the aerospace industry as leading edges on hypersonic vehicles, such as the space shuttle and X-43. Historically, these composites have been fabricated by thermosetting resins (usually phenolic based), mesophase pitches, and pyrolyzed hydrocarbon gases [1].

Bis-ortho-diynylarene (BODA)-derived resins stand as new and desirable precursor resin candidates for C/C composite manufacturing. Advantages of this resin class include high oxidative thermal stability, low flammability, and high char yield after post-carbonization (above 1000°C) [2]. These properties theoretically allow for a one step resin infiltration process, increasing the efficiency of decades-old carbon matrix precursor resin technology. As well as high char yielding, this resin system is melt-processable, rendering vacuum infiltration steps unnecessary. Current Carbon/Carbon production technology requires up to 8-9 months of processing time[3]; while using BODA-derived resins (BDR), the production process can be shortened to days. The purpose of this project, therefore, is to demonstrate the high processability of BDR by creating C/C test coupons via compression molding. Compression molding is a simplified method for creating carbon/carbon composite parts, and due to this was chosen as the manufacturing method for undergraduate engineering students.

III. Methodology

A. Materials and Mold Specifications

Our composite features a 3K 8-Harness satin weave ply and BODA-derived resin sourced from Hand Technologies, LLC. Compressive molding specimens were prepared in a Grimco Press with a 1-foot x 1-foot test bed with a student-designed mold shown in Figure 1 made of aluminum 6061-T6.

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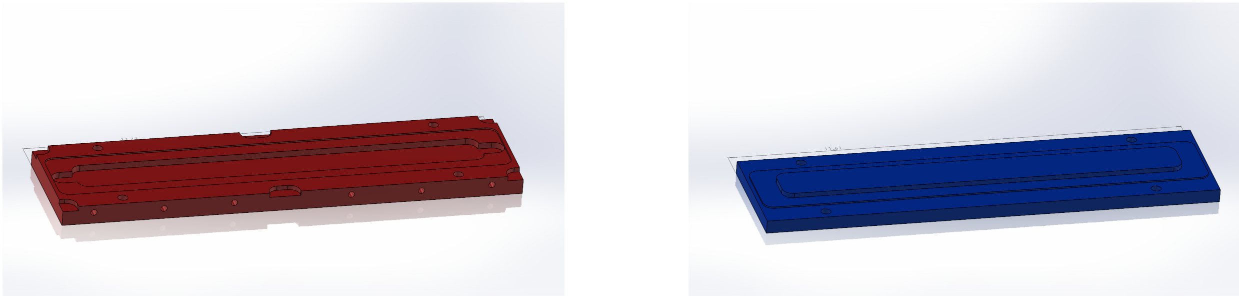

(a) SolidWorks CAD of female mold. **(b) SolidWorks CAD of male mold.**

Figure 1. Solidworks CAD of Mold

Figures 1 and 2 depict the mold and its male and female constituents with dimensions 295 mm x 76.2 mm x 21.4 mm when interlinked and is designed to create rectangular test specimens with dimensions 260 mm x 26 mm x 1 mm. Our experiments were constrained by the amount of BODA resin distributed to us by Hand Technologies LLC.; despite this, we attempted to adhere as close to the dimensions of ASTM D3039 [4], a test specification for composite tensile testing. Post-carbonized composite test articles were cut to dimensions 260 mm x 12.7 mm x 1 mm using a water saw supplied by Patterson Laboratories.

B. Layup Procedures

Within the mold, three layers of carbon fiber weaves and three layers of resin was chosen. This orientation was chosen based on the density of the carbon fiber. The density of the carbon fiber weave used is $1.76\,\mathrm{g\,cm^{-3}}$, while the density of the resin is around $1.12\,\mathrm{g\,cm^{-3}}$. With the mass from the carbon fiber, the necessary amount of resin can be calculated to achieve the necessary 60-40 mass ratio of fibers to resin. However, due to the volume of the carbon fiber weaves being unreliable to measure, individual fiber layers were cut out and then weighed in order to determine the amount of resin that needed to be used. From the bottom to the top of the mold, the layer order is as follows: fiber, resin, fiber, resin, fiber, and finally resin. This was chosen because the resin is in powder form prior to heat pressing, and by layering with the resin on top of the fiber, the resin will melt into the fiber. This is to create a more homogeneous piece and minimize delamination.

Figure 2. Compression Molding Setup

C. Compression Molding Method

To create test coupons, the procedure for compression molding is as follows: first, heat the mold to 190 degrees Celsius and hold for 15 minutes to melt the resin into the fibers. As this is occurring, the resin is partially crosslinking and causes the viscosity of the resin to increase. After 15 minutes, the compression is activated, and a force of 10 tons is applied. The temperature is brought up to 230 °C so that the coupon may fully cure, and the mold is left under isothermal compression for three hours. After this step, the process is completed, and the mold can be taken out to cool and the coupon retrieved. The mold setup in the compressive molder is seen in Figure 2.

IV. Results and Discussion

A. Coupon Manufacturing

As of date, 5 tests have been performed to create test coupons. Unfortunately, there is no prior basis to compression molding carbon matrix composites, and as such methodologies were formed based on chemical knowledge of BODA resin. Below in table 1, the 5 trials are shown and a description for each. Across all experiments, the most common and devastating issue is resin starvation and wettability. We attribute the problems of the first failures to a combination of excessive heating and miscalculated fiber ply densities.

Trial	Problem Encountered
1	Resin starved, 1 ply barely wetted
2	Resin starved, 2 ply wetted
3	Partially resin starved, successfully carbonized, upon processing catastrophically delaminated
4	No resin starvation, delamination upon carbonization
5	No resin starvation, awaiting carbonization

Table 1. Summary of trials and problems encountered.

Through trial and error, our methodologies had to change to combat the resin starvation that plagued our first trial experiments in compression molding. Our original methodology consisted of heating the open mold at 200 degrees C for 30 minutes, ramping to 230 degrees C for 30 minutes or until viscous, and then closing the mold while ramping to 250 degrees C and applying pressure. This would be held for three hours. The purpose of this methodology was to melt the resin into the plys, partially polymerize to increase viscosity (to prevent flashing of the resin when pressure was applied), and then to fully polymerize or cure into a hardened resin-fiber coupon. The mass calculations were based on the given thickness and cut out dimensions of the carbon fiber plys, as well as the density of the resin. Both the mass calculations and the compression mold schedule resulted in a failure. The carbon fiber plys were dry and loose, and appeared as if they had seen no resin at all. For the second trial, we increased the amount of resin per layer by 25%, but this trial still resulted in resin starvation.

For the third trial, the mass calculations were completely revamped. Once again, mass for resin and plys were calculated via the inner mold volume and a 60 percent mass of fiber to 40 percent mass of resin ratio. These masses represent the "ideal" conditions. Then, the individual carbon fiber plys (cut to dimension) were individually weighed out, and layers were added until the sum of their masses was just below the ideal condition. Based on a fraction of this sum to the ideal condition, then the amount of resin can be calculated by using this fraction to extract the necessary resin values. By dividing this mass by the amount of carbon fiber layers, the amount of resin per layer can be calculated. From these new values, the third trial resulted in only small amounts of visible resin starvation.

V. Future Work/Conclusions

Upon successful carbonization and processing the coupon into the proper dimensions, the coupons will immediately undergo tensile testing at ACI, as well as Scanning Electron Microscopy (SEM), and thermal cycling. We mainly desire to characterize the material properties of our composites.

This semester has been illuminating on the difficulty of processing carbon/carbon (C/C) composites. In summary, we have developed a novel methodology for compressive molding of C/C composites derived from Bis-*ortho*-Diynylarene. A student designed mold was CNC'd and milled featuring drafted beds, pop-up pieces, and pry locations. A heating schedule was then curated and improved iteratively upon completion of test coupon pieces. Our coupons will then be tensile tested and we will evaluate the material properties compared to standard C/C composites derived from phenolics. Plans to evaluate our composites under Scanning Electron Microscopy (SEM) have also been developed.

VI. Acknowledgments

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