Investigating the Fiber Content, Fiber Length, and Curing Characteristics of 3D Printed Recycled Carbon Fiber

Peng Hao Wang, Ronald Sterkenburg, Garam Kim, Yuwei He

Abstract-As composite materials continue to gain popularity in the aerospace industry; large airframe sections made out of composite materials are becoming the standard for aerospace manufacturers. However, the heavy utilization of these composite materials also increases the importance of the recycling of these composite materials. A team of Purdue University School of Aviation and Transportation Technology (SATT) faculty and students have partnered to investigate the characteristics of 3D printed recycled carbon fiber. A prototype of a 3D printed recycled carbon fiber part was provided by an industry partner and different sections of the prototype were used to create specimens. A furnace was utilized in order to remove the polymer from the specimens and the specimen's fiber content and fiber length was calculated from the remaining fibers. A differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) test was also conducted on the 3D printed recycled carbon fiber prototype in order to determine the prototype's degree of cure at different locations. The data collected from this study provided valuable information in the process improvement and understanding of 3D printed recycled carbon fiber.

Keywords-3D printed, carbon fiber, fiber content, recycling.

I. INTRODUCTION

HE early adoption of composite materials in the aerospace **I** industry was mainly focused on replacing the aircraft's secondary structures [1]. Today, as a result of the continuous advancement and research in composite material technologies, the utilization of composite materials in the aerospace industry has become standard practice. Aerospace manufacturers are now able to utilize composite materials in large and critical airframe structures instead of the traditional metal alloy materials such as the common aluminum and titanium [2]. However, as the aerospace industry push for more applications of composite materials, the increased utilization of composite materials has also resulted in the increased amounts of composite waste. Due to composite materials consisting of largely components that nature cannot decompose of naturally, the recycling of these composite materials has become increasingly crucial to the environment and aerospace industry's sustainability [3]. With many countries and business organizations now adopting the zero waste

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Ronald Sterkenburg, Garam Kim, and Yuwei He are with the School of Aviation and Transportation Technology, Purdue University, West Lafayette, IN 47907 USA (e-mail: sterkenr@purdue.edu, kim1652@purdue.edu, he273@purdue.edu). philosophy [4], it is important for the aerospace industry to investigate potential strategies for the recycling of composite materials. Currently, one of the popular strategies for recycling composite materials employs the use of 3D printing technology. 3D printing technology provides an opportunity to repurpose recycled composite fibers into other components and requires reasonably low expenses to achieve the results [5]. Therefore, together with a local industry partner, a team of faculty, and students of Purdue University's SATT teamed up with the aim of investigating the characteristics of 3D printed recycled carbon fiber.

II. METHODOLOGY

The specimens used for this study was fabricated from randomly selected sections off of the prototype 3D printed recycled carbon fiber part shown in Fig. 1. The 3D printed recycled carbon fiber part was provided by a local industry partner. These specimens were cut utilizing a diamond saw into identical 25.4 mm sections as shown in Fig. 2.



Fig. 1 Prototype 3D printed recycled carbon fiber part



Fig. 2 Specimen sectioning using a precision diamond saw

For the fiber content study, the sectioned 25.4 mm specimens were fabricated into microscopy specimens shown in Fig. 3. Specimens were placed into circular molds and filled

with clear resin. The resin was given time to fully cure before the completed microscopy specimens went through a series of polishing operations. These polishing operations help to eliminate any surface impurities and imperfections from affecting the results for the microscopic imaging of the microscopy specimens. As shown in Fig. 4, ImageJ image analyzing software was used to analyze the fiber content from the microscopic images that was taken of the microscopy specimens. was first controlled to stabilize at 30 °C. Then, the temperature was raised to 250 °C at the rate of 3 °C per minute. The chamber then soaks at 250 °C for 5 minutes before the temperature is lowered down back to 30 °C again at the rate of 3 °C per minute. Thermal analysis of the prototype 3D printed recycled carbon fiber was performed based on the data gathered from the DSC.



Fig. 3 Polished microscopy specimens



Fig. 4 Specimen microscopy imaging

The fiber length study utilized the same 25.4 mm specimens sectioned from the prototype 3D printed recycled carbon fiber part. The 25.4 mm specimens were placed in a furnace at 800 °C for a total of two hours. After two hours in the furnace, the remaining ashes shown in Fig. 5 were collected and placed on a microscope slide. Before a second microscope slide was placed on top of the ashes, a drop of acetone was applied to the ashes in order to spread the fibers within the ashes. As shown in Fig. 6, ImageJ image analyzing software was again used to analyze and measure the length of the fibers within the collected ashes.



Fig. 5 Microscope slide with ashes

The curing characteristics study of the 3D printed recycled carbon fiber was performed using DSC and DMA.

The DSC shown in Fig. 7 measures the heat flow of the tested material to analyze the thermal characteristics of the material such as glass transition temperature, phase change, crystallization, and curing kinetics. A small section of the prototype 3D printed recycled carbon fiber part was placed inside of an aluminum capsule and inserted into the DSC machine's chamber as shown in Fig. 8. The DSC's chamber



Fig. 6 Microscopic image of ashes



Fig. 7 DSC machine



Fig. 8 Specimen loaded in DSC's chamber

The DMA measures the mechanical behavior of the tested material in accordance to temperature change such as storage modulus, loss modulus, and tan delta graph. The dimensions of the specimens tested as shown in Fig. 9 were 35 mm long, 12.77 mm wide, and 3.19 mm thick. A double cantilever test fixture shown in Fig. 10 with constant amplitude temperature sweep and constant strain rate was used. The strain amplitude was 0.025 and the oscillation frequency was at 1 Hz. Temperature was ramped up to 200 °C at the rate of 5 °C per minute.



Fig. 9 DMA specimen



Fig. 10 Double cantilever test fixture for DMA

III. RESULTS

The results of the fiber content study were analyzed by the threshold method using ImageJ image analyzing software. Three specimens were analyzed by randomly selecting 50 void free areas within each of the specimens. The dimensions of the randomly selected void free areas were 500 μ m by 500 μ m as shown in Fig. 11. The average fiber content for specimen one was found to be 1.848. The average fiber content of specimen two was found to be 2.070, and the average fiber content of specimen three was 2.307.

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Fig. 11 Randomly selected void free areas in the specimens

The results of the fiber length study were again analyzed by using ImageJ image analyzing software. The collected ashes were observed under a microscope and randomly selected fibers were measured as shown in Fig. 12. A total of 2500 fibers were measured and the measurement produced an average fiber length of 47.514 μ m.



Fig. 12 Randomly selecting and measuring fiber length

The curing characteristics of the 3D printed recycled carbon fiber part was performed using DSC and DMA. The DSC thermal analysis was performed with two different specimens and analyzed separately. The glass transition temperature of the 3D printed recycled carbon fiber was found to be approximately 116 °C to 119 °C as shown in Fig. 13. The DSC results of the 3D printed recycled carbon fiber did not show resemblance to other common 3D printing media such as acrylonitrile butadiene styrene (ABS) and polylactic acid (PLA).

The DMA results as seen in Fig. 14 show the three different ways used to find the glass transition temperature of the 3D printed recycled carbon fiber. The first way was finding the glass transition temperature when the storage modulus has a large drop at 100.14 °C. Second way was finding the glass transition temperature where there is a maximum loss modulus at 106.36 °C. Lastly, the third way was finding the glass transition temperature where there is maximum tan delta at 114.66 °C. The glass transition temperature found was similar to the results of the DSC.



Fig. 13 DSC result of 3D printed recycled carbon fiber

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Fig. 14 DMA result of 3D printed recycled carbon fiber

IV. CONCLUSION

The study was able to determine the prototype 3D printed recycled carbon fiber part's fiber content, fiber length, and curing characteristics by analyzing the microscopic pictures taken of the specimens, the specimen's ashes, DSC, and DMA results.

The fiber content of the specimens was found to be relatively consistent with an average fiber content of around 2.075. However, the surprisingly low amount of fibers within the specimens poses to be a major concern. The low fiber content of the specimens suggests that the 3D printed recycled carbon fiber part will have relatively low mechanical properties due to carbon fiber's contributing factor to a component's mechanical properties [6]. Similarly, the strength of the component increases as the mean fiber length increases [7]. However, the fiber lengths within the prototype 3D printed recycled carbon fiber part were found to be inconsistent and have a very high deviation. The inconsistency and lack of control on the fiber lengths will greatly affect the component's strength predictability and resulting in the component's low reliability.

Lastly, the DSC and DMA tests were able to determine similar glass transition temperatures for the prototype 3D printed recycled carbon fiber part. The glass transition temperature was determined to be around 110 °C. However, based on the thermal analysis of the DSC and DMA results, the study was unable to determine the polymer material used for the prototype 3D printed recycled carbon fiber part. The study was able to eliminate ABS and PLA as possible polymer materials used. With the possibility of the polymer material being a blend between two different materials, further thermal analysis is required to accurately determine the exact polymer material used for the prototype 3D printed recycled carbon fiber part.

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