# CHARACTERIZATION OF THE BULK FACTOR OF AUTOCLAVE AND OUT-OF-AUTOCLAVE PREPREGS FOR SPACE APPLICATIONS

Liu, D. S.-C., Hubert, P.*

Mechanical Engineering, McGill University, Montréal, Canada
* Corresponding author (pascal.hubert@mcgill.ca)

**Keywords**: *Out-of-autoclave, space applications, debulk, thickness variation*

## 1 ABSTRACT

Out-of-Autoclave (OOA) processing of composite materials is an attractive processing method since oven acquisition and operation costs are significantly reduced compared to autoclaves. However, without autoclave pressure, OOA processing requires specific process characterization and development to ensure that high-quality panels are obtained. For space applications, the payload must withstand the harsh space environment and weight must be minimized to reduce launch costs. Thus, high performance, high quality, lightweight composite materials that can be cost effectively manufactured is highly desirable. As OOA prepregs are partially impregnated with resin, the tow impregnation during cure can cause a significant change in the laminate thickness during processing. This change in thickness, also referred to as the bulk factor, causes important variation in prepreg air permeability and large thickness deviations in corners. The objective of this paper is to measure the bulk factor of different aerospace grade prepregs. A prepreg compaction fixture was designed, where a non-contact eddy current thickness sensor monitored the laminate thickness throughout the debulk and the cure. For the modified prepreg manufacturer recommend cure cycle, results show that the thickness change during the debulk is larger compared to the cure phase. For the same fabric architecture, it was found that the bulk factor can vary significantly for different resin systems. The effect of heated debulk was investigated and it was found that a heated debulk between 48ºC and 49ºC for 2 hours resulted in similar bulk factor values as a 16-hour room temperature debulk for the same material.

## 2 INTRODUCTION

In traditional composite manufacturing, an autoclave is used. An autoclave is a pressure vessel that suppresses void formation during resin cure by applying high pressure (6.8 bars). However, autoclaves have high operating and maintenance costs, and the size of the parts are restricted to the size of the autoclave [1, 2]. Out-of-autoclave (OOA) composite processing techniques are an attractive alternative. One such technique uses only a convection oven to cure the resin and a pump connected to a vacuum bag to apply pressure. The vacuum bag effectively applies slightly less than 1 atm on the part, as perfect vacuum is impossible to achieve.

Previous research into OOA processing techniques involved creating a type of prepreg, also known as semi-preg, that has built-in air evacuation channels. Thus, thick and void free laminates were successfully made without intermittent vacuum holds prior to cure. Vacuum hold is also known as debulking. The debulk time is important for compacting the prepreg and removing entrapped air and other volatiles prior to cure. Although the details on this type of prepreg have been censored due to proprietary reasons, the concept of built-in air evacuation channels inside the prepreg remains important for OOA processing [3]. Debulking is a very time-consuming process. The amount of time

required to reduce the thickness of the laminate, thus also reducing voids, by performing room temperature debulks has been investigated. It has been shown that more than 20 hours is needed to fully debulk a plain weave prepreg [4].

The way resin in prepregs flows into dry regions during processing affects how air is evacuated and thus the void content in the final cured composite. Originally, only models and concepts existed to explain such phenomenon. One such model is the void growth model [5], an attempt to predict the possibility of void growth based on temperature, relative humidity, and resin pressure. There is also the concept of Degree of Impregnation (DOI) [6], which is the percentage of the total volume of the dry fibres that the resin has impregnated. Later, the impregnation of the dry fibre regions was experimentally investigated by examining samples taken at different points of the cure cycle with X-ray microtomography (micro-CT). It was found that the cure can affect air evacuation or void formation in the composite since prepreg air permeability decreases as the resin impregnates the fibres [7].

A successful attempt was made to reduce the debulk time through the application of heat via an infrared lamp and rapid cooling with dry ice. It was found that there is an optimal temperature at which the heating must occur, which was 65°C for CYCOM® 5320 [8]. The hot debulk temperature was selected to decrease the resin viscosity without significant cure increase during the debulk phase [9]. Using a viscosity model for CYCOM® 5320-1, debulks at 50ºC over 4 hours were recommended to replace the standard 16-hour room temperature debulk [10].

The prepreg bulk factor represents the change in material thickness during the curing phase. The change of thickness is attributed to the compaction of voids and to the fibre tow impregnation by the resin. Depending on the prepreg initial degree of impregnation and fibre architecture, OOA prepregs have a bulk factor ranging from 1.05 to 1.40. The bulk factor (β) is defined as follows:

$$\beta = \frac{t_i}{t_f} \tag{1}$$

Where, $t_f$ and $t_i$ are the final (cured) and initial (uncured) prepreg thickness respectively. It is a common practice to consider the initial prepreg thickness as the thickness after the debulking phase. The thickness can be measured with calipers, dial gauges and LVDTs. The bulk factor can be affected by the processing conditions like vacuum level, atmospheric pressure, temperature cycle and prepreg out-time. Bulk factor was identified as a major cause of thickness deviations at corners [11].

### *3.2 Objectives*

The purpose of this paper is to investigate the effect of heated debulk on autoclave and out-of-autoclave prepregs made of carbon fibre and epoxy or cyanate ester resin. Cyanate ester resins are often used in space applications since the cured resin has low moisture absorption and good thermal stability. To compare these materials, a revised version of the bulk factor is calculated. The bulk factor, β, was defined as the thickness after debulk, $t_d$, divided by the final $t_f$, thickness of the cured sample, as shown in Equation 2.

$$\beta = \frac{t_d}{t_f} \tag{2}$$

# 3 EXPERIMENTAL METHODS

## *3.2 Materials and Test Plan*

A total of five materials are investigated. This includes a combination of resins: 1) epoxy or 2) cyanate ester; intended manufacturing method: 1) autoclave or 2) out-of-autoclave; and fibre architectures: 1) 5 harness or 2) plain weave, as summarized in Table 1.

| Material | Resin | Fibre | Targeted processing method |
|---|---|---|---|
| A | Cyanate ester | 5 Harness | Autoclave |
| B | Epoxy | Plain weave | OOA |
| C | Epoxy | 5 Harness | OOA |
| D | Epoxy | 5 Harness | Autoclave/ OOA |
| E | Epoxy | 5 Harness | Autoclave |

Table 1. Materials definition

The prepreg lay-up consisted of a stack of 16 cross-ply layers, $[0/90_2/0]_{2s}$. Two debulk strategies are investigated: 1) 16-hour room temperature debulk and 2) 2-hour heated debulk between 48°C and 49°C. This temperature was determined through examining the viscosity profiles of each material. For the heated debulk experiments, an initial set-up stage of up to an hour under vacuum at room temperature was applied while all leaks in the bag were resolved. In both cases, no debulking cycles were applied during the layup process. The size of the panels was approximately 102 mm by 102 mm for the 16-hour debulk batch and 51 mm by 102 mm for the heated debulk batch.

## *3.2 Methodology*

To measure the laminate thickness and to calculate the bulk factor, an instrumented consolidation fixture, shown in Figure 1, was used inside an oven [12]. A noncontact eddy current sensor by Micro Epsilon was attached to the fixture to monitor the laminate thickness. It requires a steel target plate to function and its range is from 0.006 mm to 6 mm, with a minimum circular area of 4.2 mm. Experience has shown that a range of 1 to 4 mm is best to avoid signal noise. This sensor and the attached cable can withstand up to 150°C.

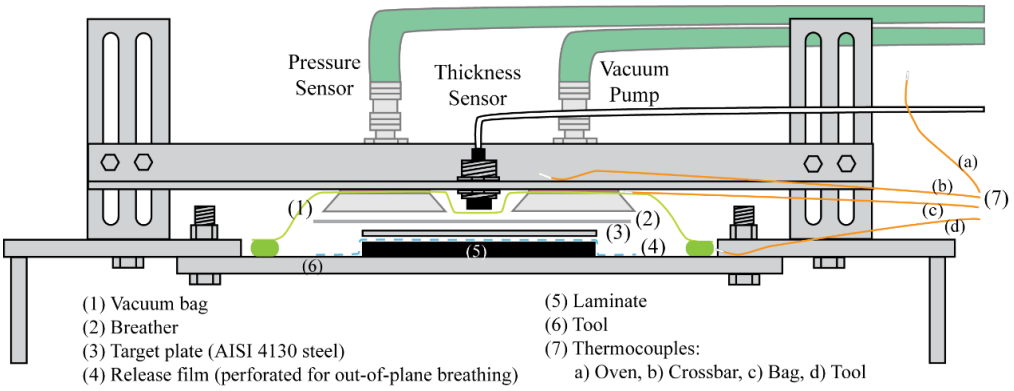


Figure 1. Bulk factor fixture.

In addition to the thickness sensor, there was also a pressure sensor attached to monitor the pressure inside the bag to ensure that the bag is not leaking. Also, a flow meter was attached along the line of the pump, outside the oven, to

monitor the flow of gases evacuated from the vacuum bag. Four OMEGA, type K, TT-K-30-SLE (RoHS) thermocouples were used to monitor the oven, crossbar, bag, and tool temperature. The temperature of the crossbar is especially important since it is used to calculate the laminate displacement. Polynomial conversion curves were experimentally found for several temperatures and used in conjunction with the crossbar temperature to convert the sensor signal to millimetres. The calibration curves not only consider for the expansion of the fixture and target plate, but also the non-linearity of the eddy current sensor's readings at high temperatures.

The laminate final cured thickness was measured with a digital Vernier caliper. The laminate thickness during the debulk and curing process was computed by adding the difference between the final and actual displacement reading to the laminate final cured thickness.

The cure cycles used are listed in Table 2, for the a) 16-hour room temperature debulk (RTD) and the b) heated debulk (HD).

| **Cure Cycle** | **Cure Cycle Steps** | **Materials** |
|---|---|---|
| Debulk | Hold 16 hours at RT | A, B, C, D, E |
| 0 | Ramp to 135°C at 3°C/min<br>Hold *<br>Cool to RT | |
| * | 180 min<br>360 min<br>360 min<br>1510 min<br>120 min | A<br>B<br>C<br>D<br>E |

a)

| **Cure Cycle** | **Steps** | **Materials** |
|---|---|---|
| Debulk | Ramp to 48°C at 1°C/min<br>Hold 120 min | A, B, C, D, E |
| 1 | Ramp to 84°C at 1°C/min<br>Hold 300 min<br>Ramp to 139°C at 1°C/min<br>Hold 130 min<br>Cool to RT | A, E |
| 2 | Ramp to 108°C at 1°C/min<br>Hold 120 min<br>Ramp to 144°C at 1°C/min<br>Hold 205 min<br>Cool to RT | B, C, D |

b)

Table 2. a) Room temperature debulk (RTD) and b) heated debulk (HD) cure cycle definitions

Cure kinetic simulations for each of the material models [13] were used to develop the cure cycles listed in Table 2. The models confirmed that the heated debulk advanced the material cure by less than 0.0013. The final degree of cure prediction ranged from 0.7 for material D to 0.9 for materials A, B, C, and E.

Additionally, micro CT scans were taken for one ply of each material in their uncured state.

# 4 RESULTS

Through examining the panels after manufacturing, different levels of resin bleeding could be identified. Materials A, B, C and E showed no or very little signs of resin bleeding. Material D showed significant amount of resin bleeding. This was attributed to the lower minimum viscosity of material D.

For room temperature debulk (Figure 2), 60 to 80% of the thickness change occurred within 5 minutes of the room temperature debulk phase. For heated debulk (Figure 3), a similar trend was observed where 61 to 82% of the thickness change occurred by the start of the heated debulk hold.

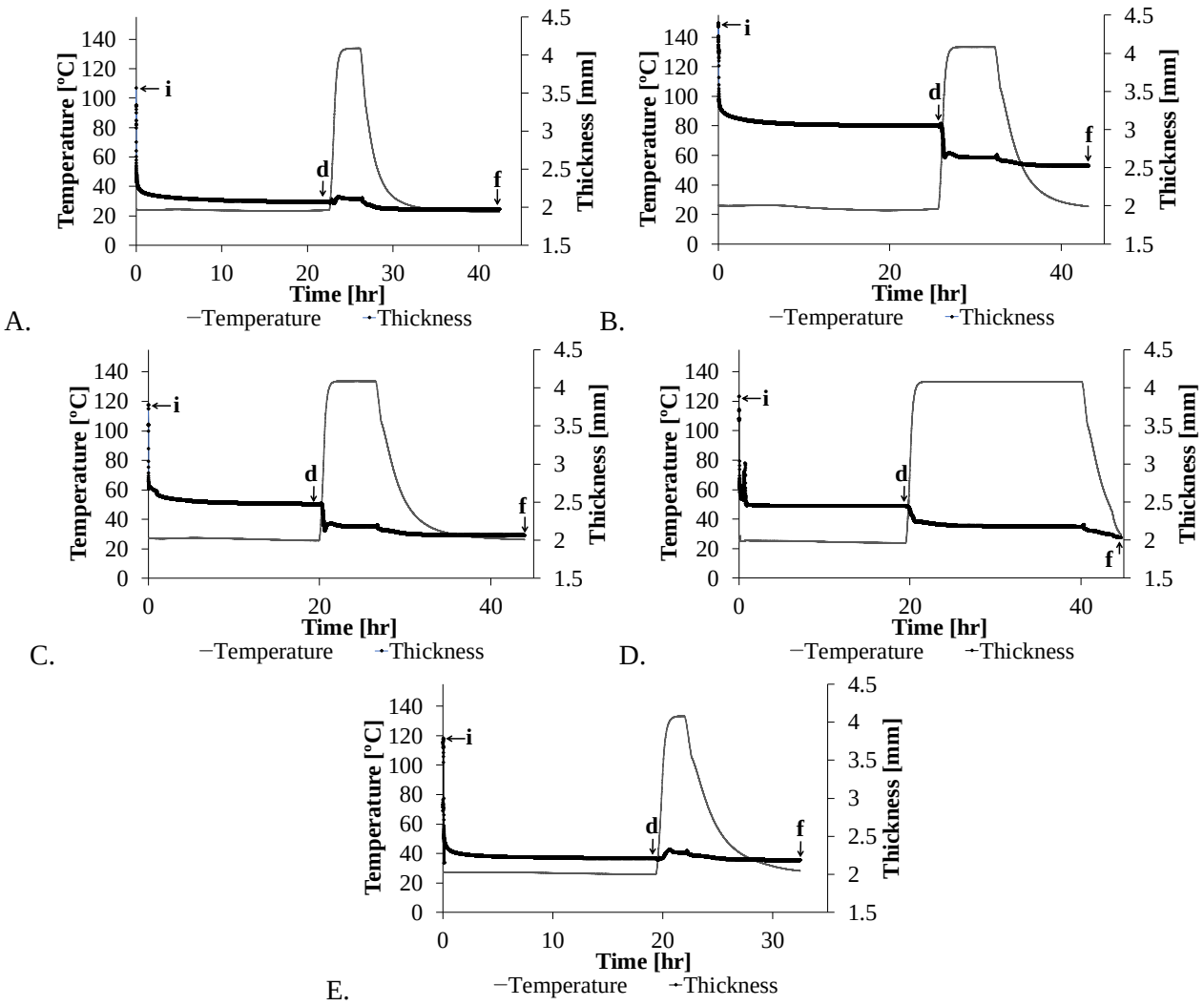


Figure 2. Plot of change of thickness over crossbar temperature for materials A to E, 16-hour room temperature debulk

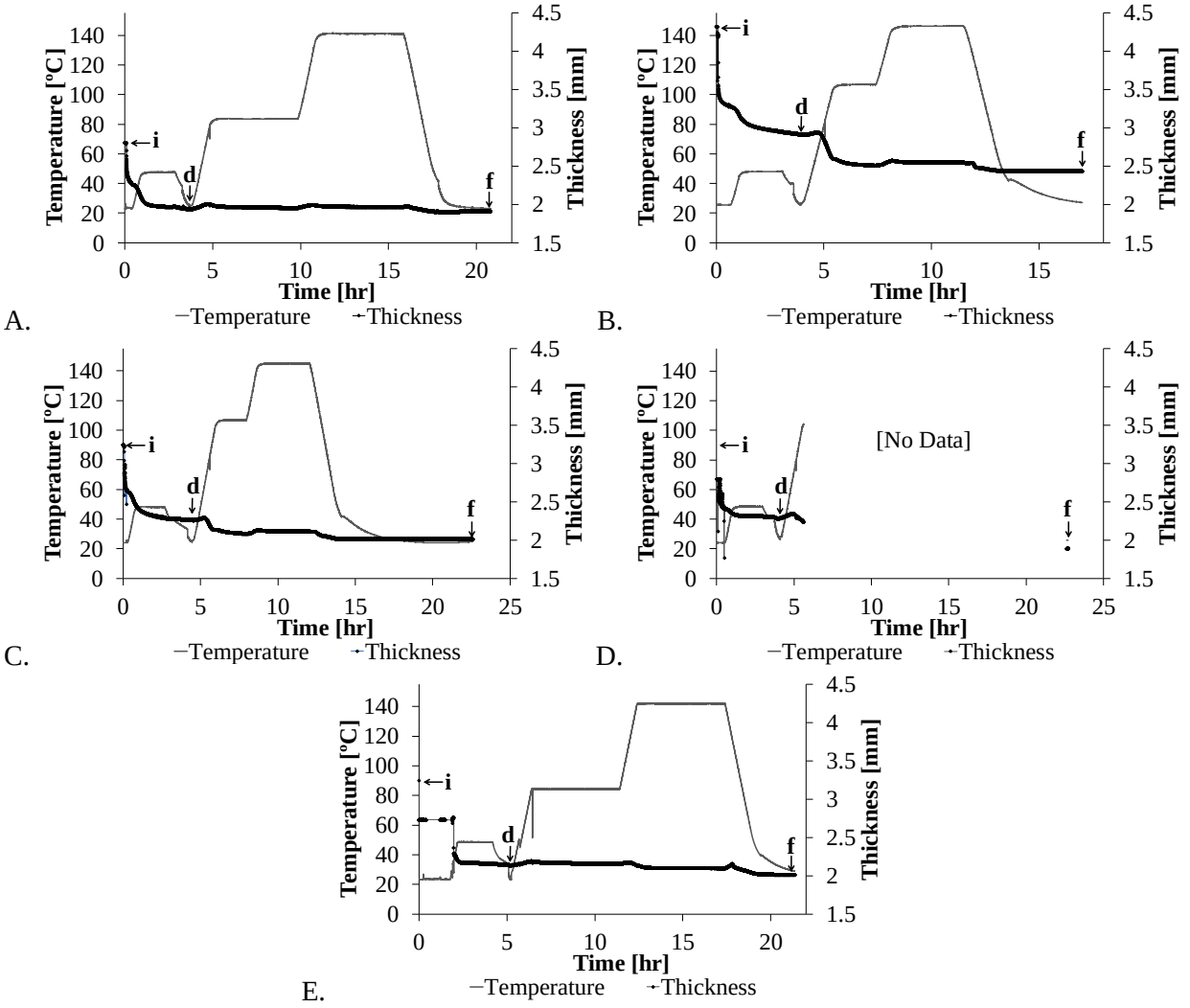


Figure 3. Plot of change of thickness over temperature for materials A to E, heated debulk

Figure 4, summarizes the thickness change during cure: the final cured thickness and the thickness change due to debulk ($\Delta_{debulk}$) and cure ($\Delta_{cure}$). The final cured thickness of all materials was around 2 mm, except for material B, which was thicker since it contained a plain weave fabric. Also, the thickness change due to cure ($\Delta_{cure}$) was similar for all materials regardless of cure cycle followed.

In Figure 5, the bulk factor values were calculated per Equation 2. For each material, despite undergoing a different debulking process and cure cycle, the bulk factor values averaged to 1.0 for materials A and E, materials made for use in an autoclave, and 1.2 for materials B, C, and D. Materials B and C were made for out-of-autoclave use and material D was made for both autoclave and out-of-autoclave use.

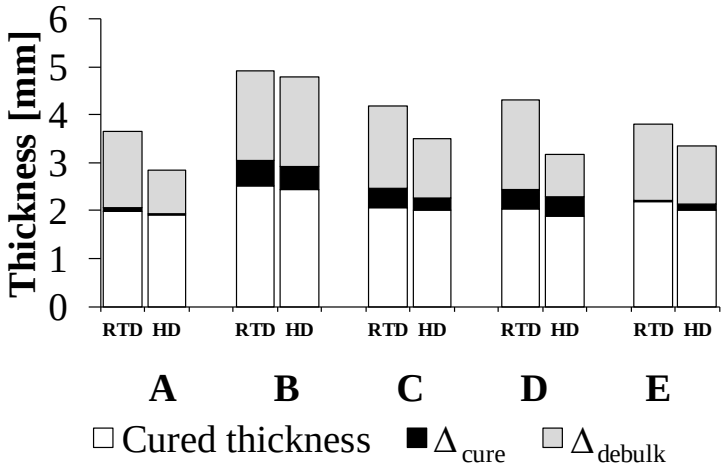


Figure 2. Cured thickness and change in thickness due to debulk ($\Delta_{debulk}$) and cure ($\Delta_{cure}$)

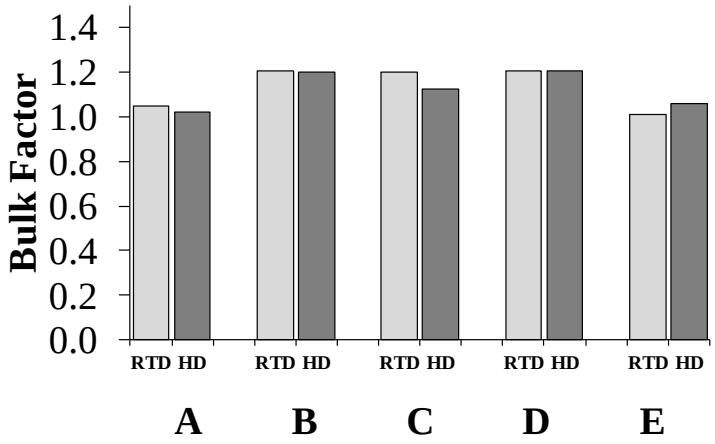


Figure 3. Bulk factor values for room temperature debulk (RTD) and heated debulk (HD)

Figure 6 shows the results of the micro CT scans of one layer of each uncured material. Material B looks very different from the other materials due to a different weave, plain weave instead of 5 harness. The weave has a larger bundle of tows and larger holes between tows. The resulting side view shows an overall thicker prepreg with symmetrical undulations, whereas the 5 harness is thinner and looks more like a sine or cosine wave. This explains why this material is thicker than the other materials when cured. It also has a large change in thickness due to debulk and cure. There is a relatively even spread of resin (bright white) all over materials A and E. These are autoclave materials, which showed no resin bleeding and had a bulk factor that averaged to 1.0. Meanwhile, the resin in materials B, C, and D seem to be unevenly distributed, with the bright white areas at different spots. A significant amount of resin is coating material D, an autoclave/ out-of-autoclave material, which showed the most resin bleeding. Materials B and C are out-of-autoclave materials. They have a porous structure that allows for air evacuation, typical

of semi-pregs, thus also a bulk factor that averaged to 1.2. From these images, given such large structural differences, it can be seen why the bulk factor would be different despite of having the same fabric architecture.

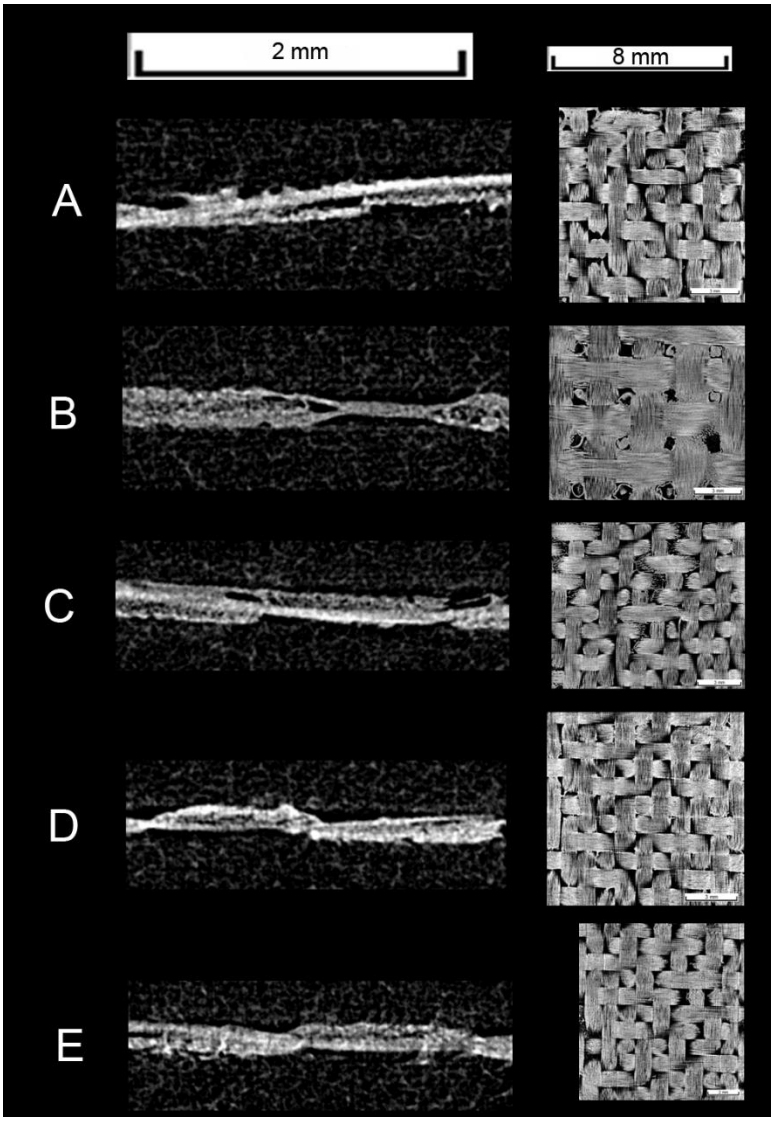


Figure 6. Micro CT scans of the five uncured materials

Figure 7 is a summary of the total cycle time (debulk and cure) for each material, for 16-hour and heated debulks. Without a doubt, heated debulk is a much more attractive choice because the cycle time is reduced. By following the heated debulk cycle, from 26% to 71% of the original 16-hour cycle time can be removed. This means that more parts can per produced given the same amount of time.

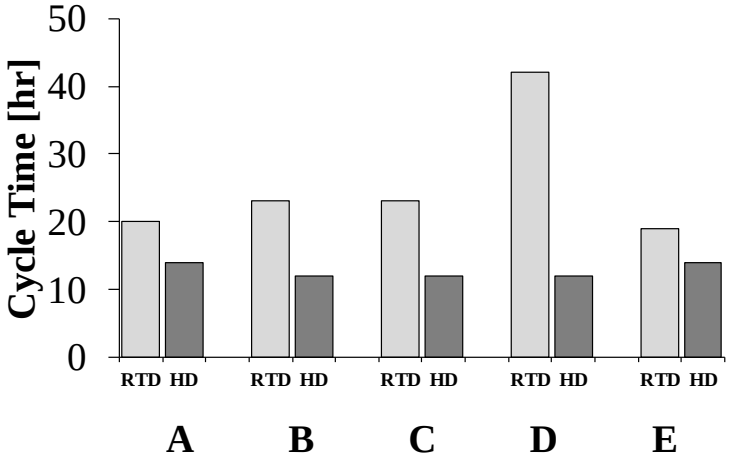


Figure 7. 16-hour debulk (RTD) vs. heated debulk (HD) cycle times for each material

# 5 CONCLUSIONS

A 2-hour heated debulk between 48 and 49 ºC can replace a 16-hour room temperature debulk. The resulting bulk factor values averaged to 1.0 for materials made for use in an autoclave and 1.2 for out-of-autoclave. This applies to both debulk cycles and all materials studied, which are a combination of epoxy and cyanate ester resins, 5 harness and plain weave fabric architectures, and autoclave and/or out-of-autoclave targeted processing methods. Heated debulk reduces the cycle time by 26% to 71%, which makes it a very attractive processing method. Inspecting micro CT images of the uncured materials show structural differences that explain differences in bulk factor values of materials with the same fabric architecture.

# 6 ACKNOWLEDGEMENTS

This research has been conducted with financial and/or material support from MDA Corporation, Stelia Aerospace, the Consortium for Research and Innovation in Aerospace in Québec (CRIAQ), the National Sciences and Engineering Research Council of Canada (NSERC), the Research Centre for High Performance Polymer and Composite Systems (CREPEC), and the McGill Engineering Doctorial Awards (MEDA). The authors would like to acknowledge Master's student Julieta Barroeta Robles and summer students Camille Rousseau and Veronica Shaheen for performing the resin characterization experiments and/or developing the resin cure kinetics and viscosity models, which were used to find the cure cycles, and manufacturing the panels with a 16-hour debulk. Also, the authors would like to thank Research Assistant Lucie Riffard for her constant support and helping with performing the resin characterization experiments.

# 7 REFERENCES

[1] K. Jackson, M. Crabtree. "Autoclave Quality Composites Tooling for Composite from Vacuum Bag Only Processing*". 47th Int. SAMPE Symposium Exhib*., vol. 47, pp 800–807, 2002.

[2] S. G. Advani, K.-T. Hsiao. "*Manufacturing Techniques for Polymer Matrix Composites (PMCs)*". Philadelphia, PA, Woodhead Pub., 2012.

[3] B. Thorfinnson. "Production of Void Free Composite Parts without Debulking". *Proc. 31st Int. SAMPE Symp*., Las Vegas, NV, pp 480–490, 1986.

[4] C. Ridgard. "Out of Autoclave Composite Technology for Aerospace, Defense and Space Structures". *SAMPE 09 Spring Symp. Conf. Proc*., Baltimore, MD, United States, 2009.

[5] J. Kardos, M. P. Duduković, R. Dave. "Void Growth and Resin Transport during Processing of Thermosetting-matrix Composites". *Adv Polym Sci*., vol. 80, pp 101–123, 1980.

[6] B. Thorfinnson. "Degree of Impregnation of Prepregs - Effects on Porosity". *Proc. 32nd Int. SAMPE Symp*., Anaheim, CA, pp 1500–1509, 1987.

[7] T. Centea, P. Hubert. "Measuring the Impregnation of an Out-of-Autoclave Prepreg by Micro-CT". *Compos Sci Technology*, vol. 71, pp 593–599, 2011.

[8] S.-M. Hughes. "Thermally Assisted Consolidation of Out-of-Autoclave Prepreg for Concave Parts". Master's Thesis. *McGill University*, 2016.

[9] J. Kratz, K. Hsiao, G. Fernlund, P. Hubert. "Thermal Models for MTM45-1 and Cycom 5320 Out-of-Autoclave Prepreg Resins". *J Compos Mater*, vol. 47, pp 341–352, 2013.

[10] C. Ridgard. "Process Selection and Optimization for Out-of-Autoclave Prepreg Structures". *SAMPE J*., vol. 52, pp 7–15, 2016.

[11] A. Levy, J. Stadlin, P. Hubert. "Corner Consolidation in Vacuum Bag Only Processing of Out-of-Autoclave Composite Prepregs Laminates". *Proc. SAMPE Tech. Conf*., Seattle, WA, 2014.

[12] T. Centea. "Material - Processing - Quality Relationships during the Consolidation of Out-of-Autoclave Prepregs". PhD Thesis. *McGill University*, 2013.

[13] J. Barroeta Robles, C. Rousseau, P. Hubert. "Material Characterization to Evaluate the Suitability of Out-of-Autoclave Semipregs for Structural Space Applications". *10th Canadian-International Conference on Composites*, Ottawa, Canada, July 17-20, 2017.