ARMY RESEARCH LABORATORY

Tailoring Fiber Volume Fraction of Vacuum-assisted Resin Transfer Molding Processed Composite Laminates by Bladder-bag Resin Reservoir

by Zachary J. Larimore and Larry R. Holmes, Jr.

ARL-TN-0510 **November 2012**

NOTICES

Disclaimers

Army Research Laboratory

Aberdeen Proving Ground, MD 21005-5069

ARL-TN-0510 **November 2012**

Tailoring Fiber Volume Fraction of Vacuum-assisted Resin Transfer Molding Processed Composite Laminates by Bladder-bag Resin Reservoir

Zachary J. Larimore
Oak Ridge Institute for Science and Education

Larry R. Holmes, Jr.
Weapons and Materials Research Directorate, ARL

REPORT DOCUMENTATION PAGE	*Form Approved* *OMB No. 0704-0188*

1. REPORT DATE *(DD-MM-YYYY)* November 2012	**2. REPORT TYPE** Final	**3. DATES COVERED (From - To)** January 2012

4. TITLE AND SUBTITLE Tailoring Fiber Volume Fraction of Vacuum-assisted Resin Transfer Molding Processed Composite Laminates by Bladder-bag Resin Reservoir	**5a. CONTRACT NUMBER** **5b. GRANT NUMBER** **5c. PROGRAM ELEMENT NUMBER**
6. AUTHOR(S) Zachary J. Larimore* and Larry R. Holmes, Jr.	**5d. PROJECT NUMBER** MMCP04B **5e. TASK NUMBER** **5f. WORK UNIT NUMBER**
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Research Laboratory ATTN: RDRL-WMM-A Aberdeen Proving Ground, MD 21005-5069	**8. PERFORMING ORGANIZATION REPORT NUMBER** ARL-TN-0510
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	**10. SPONSOR/MONITOR'S ACRONYM(S)** **11. SPONSOR/MONITOR'S REPORT NUMBER(S)**

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

* Oak Ridge Institute for Science and Education

14. ABSTRACT

Classical composite laminated plate theory (LPT) shows that high fiber volume fraction (fvf) composites in the range of 0.60 to 0.65 can optimize the strength to weight ratio of composite structure. Current processing methods capable of achieving idealized fvf use a high-pressure autoclave and require pre-impregnated fibers with specialized resin volume. Autoclave processing is extremely high cost due to inherently long cycle time, specialized non-portable equipment, and high-cost molds. Structural requirements of aerospace applications serve as justification for the high cost of autoclave processes. The Vacuum-Assisted Resin Transfer Molding (VARTM) process is an alternative, out-of-autoclave, method which is attractive due to its low cost, simplicity, and portability. However, a limitation to current VARTM processing methods is the ability to tailor, or increase, the fvf of the composite laminate. Reducing resin volume available to the optimum resin volume, calculated using LPT, can yield increased fvf in the composite laminate while preserving complete wet-out of the fiber plies. Pre-measured volumes of resin placed in a bladder bag within the consolidation vacuum bag allow for the fvf of the composite to be controlled by limiting the resin volume available to flow into the fiber. This processing technique also has potential as a small scale field repair option.

15. SUBJECT TERMS

out of autoclave, composite processing, composite fabrication, VARTM

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON** Larry R. Holmes
a. REPORT Unclassified	**b. ABSTRACT** Unclassified	**c. THIS PAGE** Unclassified	UU	16	**19b. TELEPHONE NUMBER** (*Include area code*) 410-306-4951

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

Contents

List of Figures

List of Tables

Acknowledgments

The author would like to thank the Oak Ridge Institute for Science and Education (ORISE) for their support.

The author would also like to acknowledge the technician staff in the Composites Processing Laboratory for their advice on composites processing techniques throughout the initial development of this process.

INTENTIONALLY LEFT BLANK.

1. Introduction

There is a large demand for processes that can produce high fiber volume fraction (fvf) composites at a low cost. This is because high fvf composite structures can have greater strength-to-weight ratios than their lower fvf counterparts. Pre-impregnated (pre-preg) fibers in an autoclave process are capable of achieving high fvf, in large part, due to the tailored resin volume available to the system and because of the high-pressure capabilities of the autoclave. However, autoclaves are high-expense systems with steep operating costs. Pre-preg fiber systems also require cold storage and have significantly shorter shelf lives than two part epoxy systems (*1–6*). Vacuum-Assisted Resin Transfer Molding (VARTM) is a common low cost alternative to autoclave processing due to its simplicity, low cost, and portability (*7*). However, during traditional VARTM processing, the fiber system is infiltrated from an excess resin reservoir until complete wet-out, limiting tailorability of the resin volume transferred to the pre-form. This lack of process control typically prevents VARTM from achieving a fvf greater than 0.50. Bladder-Bag VARTM (BBVARTM) infusion endeavors to recreate the tailored resin volume available to the pre-form in pre-preg systems, while maintaining the low cost and portability of a traditional VARTM process.

In pre-preg systems, the volume of resin supplied to the pre-form through impregnation of the fibers is calculated using laminated plate theory (LPT) to yield a precise fvf upon curing (*1–6*). In the same fashion, the amount of resin to be included in the bladder-bag system can be calculated to supply the pre-form with the exact amount of resin required for a pre-determined fvf. By placing the desired amount of resin directly into the consolidation vacuum bag, the lack of tailorability associated with traditional VARTM infiltration is addressed.

2. Experimental

Six-inch square panels were produced from 24 oz S-2 fiberglass with SC-15 toughened epoxy as the resin system. The volume of resin in each bladder bag was calculated using LPT. Panels were fabricated with resin content available to the system to produce panels with a fvf ranging from 0.35 to 0.60 in 0.05 increments. When attempting to make high fvf panels with the modified VARTM process, it has been common to encounter higher void content due to the decreased resin content (*8*). Therefore, low fvf panels were fabricated to demonstrate if this novel process is capable of producing high-quality parts, i.e., low-void content.

2.1 Calculations

The amount of resin required to yield a given fvf was calculated by first determining the density of the resulting composite using equation 1:

$$\rho_c = \rho_f V_f + \rho_m V_m \tag{1}$$

Where ρ_c, ρ_f, and ρ_m are the densities of the composite, fiber, and matrix, respectively; and V_f and V_m are the desired fiber and matrix volume fractions, respectively. The weight fraction of the matrix was then calculated using equation 2; where W_m is the matrix weight fraction:

$$W_m = \frac{\rho_m}{\rho_c} V_m \tag{2}$$

The overall weight of the composite was then calculated by multiplying the volume of the composite by the density of the composite. Finally, the weight of the resin required was calculated by multiplying the matrix weight fraction by the weight of the composite (*9*).

2.2 Methods

To prepare the bladder bags: (1) SC-15 resin was mixed to the manufacturer's standards, (2) the mixed resin was then degassed in a vacuum oven at ~28 in of mercury for 30 min, and (3) the resin was then transferred into vacuum-sealable storage bags (manufactured by FoodSaver). The bags were then sealed and all air was evacuated from the bag through the vacuum port, eliminating the introduction of voids into the pre-form via air bubbles within the resin bag. The resin was then sealed in the lowest volume of bag necessary, using a heat sealer. This was done to prevent having excess bladder-bag material within the consolidation vacuum bag; limiting the potential for trapped resin, which could prevent the cured composite from meeting the predicted fvf. This can be seen in figure 1.

Figure 1. Vacuum storage bag, vacuum sealer, and heat-sealed packet of resin.

The 6 in square panels were then bagged in the same fashion as a typical VARTM lay-up, figure 2, with the only exception that the resin inlet tube was replaced with the bladder bag. Figure 3 shows a BBVARTM lay-up immediately following the initiation of resin infusion. Following initial testing, it was determined that there was a need for a device inside of the consolidation bag to rupture the bladder bag and initiate resin flow. It was necessary that the device present no threat to the integrity of the vacuum bag or the tool/part while still creating an adequate opening to allow efficient infusion of the pre-form. To address this need, a piece of polycarbonate was laser-machined to have serrations and then bent. This device was then placed on top of the bladder bag inside of the consolidation bag with the teeth angled down (toward the tool surface). This allowed for the easy initiation of flow by pressing the serrations through the bladder bag while presenting no threat to the integrity of the vacuum bag due to the bend in the device keeping the teeth in contact with the tool surface after rupture of the bag. The infusion initiation device can be seen in figure 4.

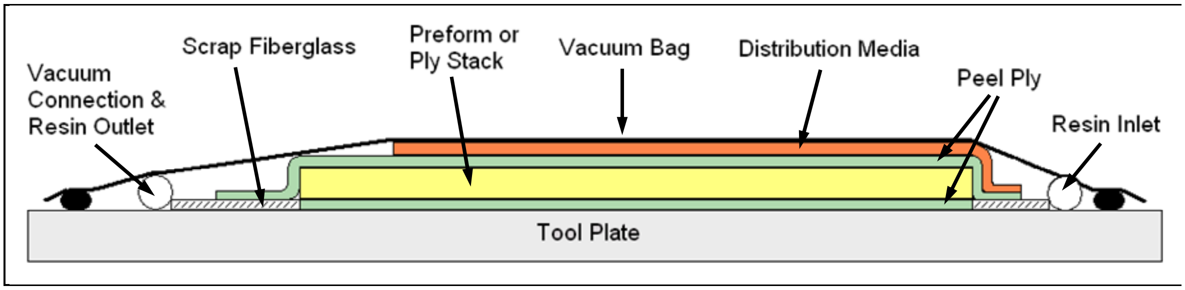

Figure 2. Typical VARTM lay-up.

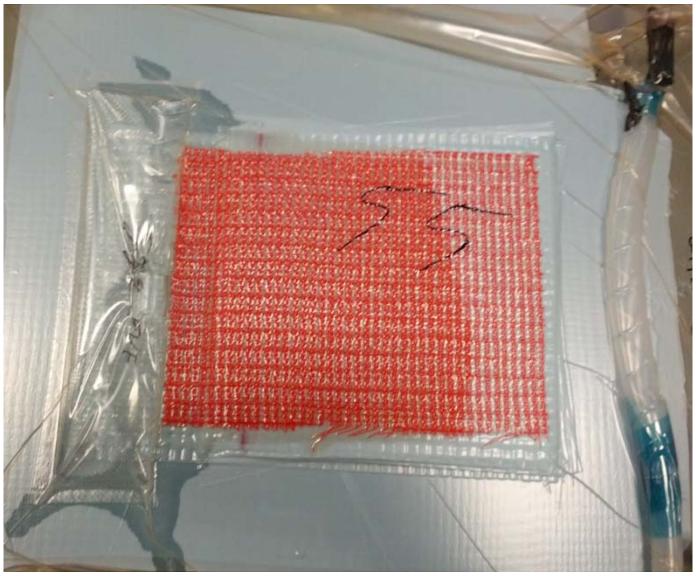

Figure 3. BBVARTM lay-up after initiation of resin infusion.

Figure 4. Resin bladder bag with infusion initiation device.

After full wet-out, the panels were kept under vacuum overnight and then post-cured the following day at 250 °F. Four samples were cut from each panel (one from each quadrant of the panel at least 1 in from the edges of the panel). The fvf of each sample was then determined following ASTM D-2584 for the ignition loss of cured reinforced composites (*10*).

3. Results and Discussions

Two panels were tested at each fabricated fvf. The results of the ignition loss test are tabulated in table 1 and the average actual fiber volume, resin volume, and void volume for the specimen has been plotted in figure 5.

Table 1. Results of burnout test.

Predicted fvf (%)	**Actual**		
	V_f (%)	**V_r (%)**	**V_v (%)**
35	47.8 (s = 1.0)	50.9 (s = 1.0)	1.3 (s = 0.0)
40	48.9 (s = 0.8)	48.3 (s = 0.9)	2.8 (s = 0.1)
45	52.1 (s = 0.4)	45.9 (s = 1.0)	2.0 (s = 0.8)
50	52.8 (s = 1.1)	45.5 (s = 1.2)	1.7 (s = 0.7)
55	54.6 (s = 0.4)	43.4 (s = 0.5)	2.0 (s = 0.4)
60	53.2 (s = 1.7)	42.1 (s = 3.6)	4.7 (s = 2.8)

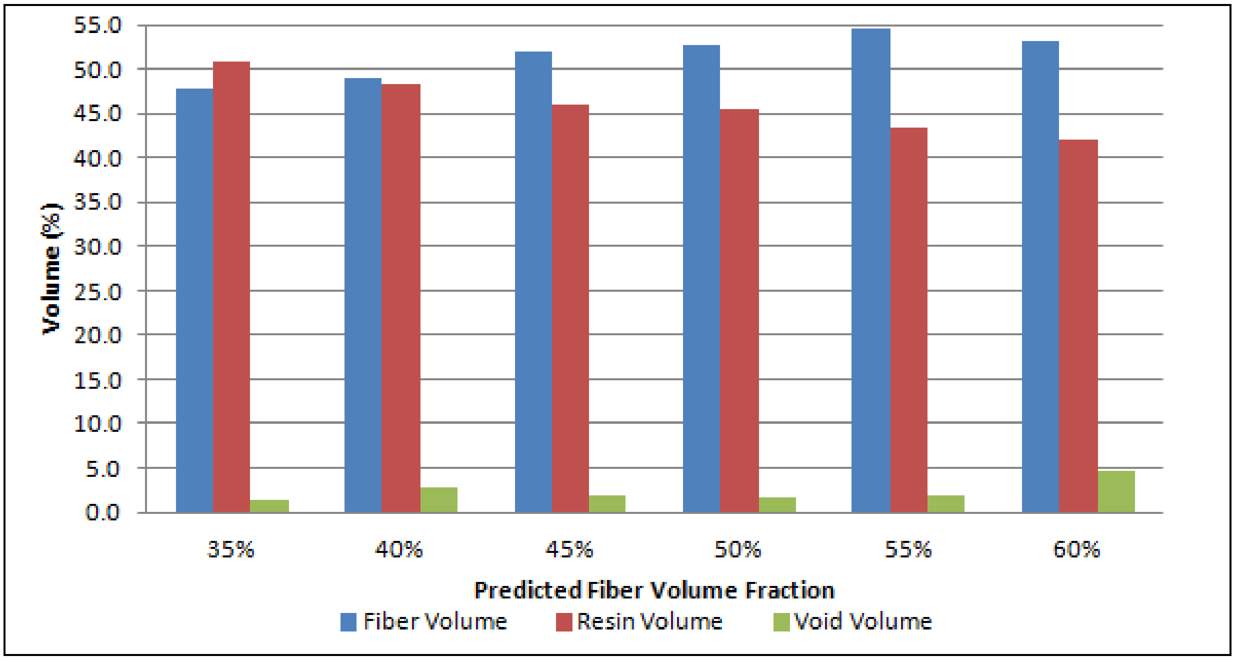

Figure 5. Plot of fiber, resin, and void volume fractions for each specimen.

It can be seen from the data that fvf was not accurately controlled. However, upon further inspection, it can be noted that the data contains an upward trend in actual fiber volume versus the predicted fiber volume fraction, with the exception being the attempts at 60%. The examination of the data at the 60% samples shows a significantly increased void fraction and a drop in fvf from the 55% specimen. This likely occurs because of the lack of resin available to the pre-form preventing complete wet-out of the fiber. If the samples at 60% are excluded, the data for fvf fits a linear trendline with an R-squared value of 0.96. Therefore, while not matching the predicted fvf from LPT; fvf was controlled by varying the resin available to the pre-form. It is expected that the fvf was not accurately controlled at the lower fiber volume fractions because the pressure supplied to the pre-form by the consolidation bag will not allow lower fvf to be readily achieved.

From further trials, an equation for accurately tailoring fvf of composites through bladder-bag resin reservoir could be formulated. A typical traditional VARTM part has an fvf of 0.50 with a void content below 3% (*11*). Therefore, this method demonstrates the ability to produce high-quality composite parts with a nearly 10% increase in fiber volume over traditional VARTM processes. Further testing and development of this system must be carried out to determine if the mechanical strength of the laminates produced with this method meet the standards of traditional VARTM processes. Also, it must be determined if this process applicable to other fiber/resin systems, as well as if it is scalable to larger laminate structures. Further optimization and testing of this processing technique will be carried out to address these issues, as well as to gather adequate data for the development of a formula for accurately tailoring fvf of BBVARTM laminates.

4. References

1. Li, X.; Carlsson, L.; Davies, P. Influence of Fiber Volume Fraction on Mode III Interlaminar Fracture Toughness of Glass/Epoxy Composites. *Composites Science and Technology* **2004**, *64* (9), 1279–1286.

2. Davies, P.; Casari, P.; Carlsson, L. Influence of Fiber Volume Fraction on Mode II Interlaminar Fracture Toughness of Glass/Epoxy Using the 4ENF Specimen. *Composites Science and Technology* **2005**, *65* (2), 295–300.

3. Hockin, H. et al. Effects of Fiber Volume Fraction on Mechanical Properties of SiC-Fiber/Si3N4-Matrix Composites. *Journal of the American Ceramic Society* **1994**, *77* (7), 1897–1900.

4. Okoli, O.; Smith, G. The Effect of Strain Rate and Fibre Content on the Poisson's Ration of Glass/Epoxy Composites. *Composite Structures* **2000**, *43*, Issues 1–3, 157–161.

5. Gu, W.; Wu, H.; Kampe, S.; Lu, G. Volume Fraction Effects on Interfacial Adhesion Strength of Glass-Fiber-Reinforced Polymer Composites. *Materials Science and Engineering: A* **2000** *277*, Issue 1–2, 237–243.

6. Mason, K. Autoclave Quality Outside The Autoclave? *High-Performance Composites* **2006**.

7. Lewit, S.; Jakubowski, J. Low Cost VARTM Process for Commercial and Military Applications. Proceedings of the *42nd International SAMPE Symposium*, Anaheim, CA, 1997, pp 1173–1187.

8. Holmes, L.; Wolbert, J.; Gardner, J. *A Method for Out-of-Autoclave Fabrication of High Fiber Volume Fraction Fiber Reinforced Polymer Composites*; ARL-TR-6057; U.S. Army Research Laboratory: Aberdeen Proving Ground, MD, 2012.

9. Bhagwan D.; Agarwal; Broutman; L. J.; Chandrashekhara, K. *Analysis and Performance of Fiber Composites*, John Wiley and Sons, Inc., 2006.

10. ASTM D-2584. Standard Test Method for Ignition Loss of Cured Reinforced Resin, American Society for Testing and Materials, *Annu. Book ASTM Stand*. West Conshohocken, PA, **2011**.

11. Heider, D., Gillespie, J. VARTM Variability and Substantiation. *Proceedings of The Joint Advanced Materials and Structures (JAMS) Center of Excellence*, University of Washington, June 2010.

List of Symbols, Abbreviations, and Acronyms

BBVARTM	Bladder Bage Vacuum-Assisted Resin Transfer Molding
fvf	fiber volume fraction
LPT	laminated plate theory
ORISE	Oak Ridge Institute for Science and Education
VARTM	Vacuum-Assisted Resin Transfer Molding

NO. OF COPIES	ORGANIZATION
1 (PDF only)	DEFENSE TECHNICAL INFORMATION CTR DTIC OCA 8725 JOHN J KINGMAN RD STE 0944 FORT BELVOIR VA 22060-6218
1	DIRECTOR US ARMY RESEARCH LAB IMAL HRA 2800 POWDER MILL RD ADELPHI MD 20783-1197
1	DIRECTOR US ARMY RESEARCH LAB RDRL CIO LL 2800 POWDER MILL RD ADELPHI MD 20783-1197
1	DIRECTOR US ARMY RESEARCH LAB RDRL CIO LT 2800 POWDER MILL RD ADELPHI MD 20783-1197

ABERDEEN PROVING GROUND

5	RDRL WMM A J WOLBERT J GARDNER Z LARIMORE L HOLMES JR (2 COPIES) 4600 DEER CREEK LOOP ABERDEEN PROVING GROUND MD 21005