Effects of Bleeder Schedule to Fiber Mass Fraction and Composites Surface Topography on Wet Lay-up Manufacturing Process

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Abstract

The thermal protector materials of the rocket's motor are made by a wet lay-up manufacturing process. Since the fiber mass fraction of the product is low, several experiments have been conducted to solve this problem including changing the type of the epoxy resin and selecting the most suitable bleeder schedule. Bleeder cloth application results in increasing the fiber mass fraction. The fiber mass fraction of thermal protector material manufactured by hand lay-up can reach a maximum of 56.78%, whereas vacuum bagging can reach a maximum of 66.43%. Peel ply and breather fabric combination are the best bleeder schedule for the hand lay-up method meanwhile perforated release film and breather fabric are the best bleeders for the vacuum bagging method. Composite surface topography obtained from peel ply is visible on the surface. The imprints of the nylon peel ply weave are visible through SEM analysis. Meanwhile, the surface topography obtained from the perforated release film is not visible. The vacuum bagging method helped reduce the number of voids and ductile polymer fractures from the composite surface. This paper recommends peel ply usage in the thermal protector manufacturing process to replace the sanding or filling method that the author use nowadays.

Keywords: *thermal protector material, bleeder schedule, fiber mass fraction, SEM analysis, peel ply.*

Nomenclature

X_f	=	fiber mass fraction
m_f	=	fiber mass, g
m_t	=	composite total mass, g
A_f	=	fiber area, cm^2
F_{aw}	=	fiber areal weight, $g/_{cm^2}$

1. Introduction

LAPAN's rocket motor thermal protector materials are made of fiberglass and fiber carbon epoxy composites. The materials are manufactured by the wet lay-up process. Wet lay-up is a method of making a reinforced product by applying a liquid resin system while or after the reinforcement is put in place (U.S. Departement of Defense, 2002). The resin and fiber are applied to the working surface by hand. The fiber mass fraction of the composites product made by wet lay-up is low (less than 50%). The thermal protector materials are rich in polymer resin. Experiments are conducted to increase the fiber mass fraction. Since the manufacturing process cannot be replaced, there are efforts to replace the type of epoxy resin and to apply the bleeder cloth during the manufacturing process. Change in the type of epoxy will be described in another work. This work is focused on the bleeder type effect on the fiber fraction and composite surface laminate.

Bleeder cloth is a nonstructural layer of material used in the manufacture of composite parts to allow the escape of excess gas and resin during cure. The bleeder cloth is removed after the curing process and is not part of the final composite (U.S. Departement of Defense, 2002). During the manufacturing process, the excess polymer resins are adsorbed by the bleeder cloth, increasing the fiber fraction of the composites product.

There are many types of bleeder: peel ply fabric, perforated release film, woven fabric, breather fabric, etc. Peel ply and perforated release film are often used as the first bleeder facing the laminates. Woven fabric and breather fabric are put upon the peel ply or perforated release film. Peel ply, the most popular one, is an extra layer of fabric material that is laid upon the outer surface of the composite during fabrication (Raymond F. Wegman, 2013). Peel ply fabric is usually made of nylon or polyester. Meanwhile, the perforated release film is a thin layer of plastic film with pores to control the flow of the excess resin during the early stages of the curing process. Perforated release films are made of polyethylene or HDPE. Peel ply can adsorb the excess resin but the perforated release film cannot. Therefore, the difference between bleeder properties gives a unique texture to the surface of the laminates.

2. Methodology

2.1. Related Works

The optimization of the lining system for solid rocket motors has been conducted multiple times. Sutrisno in 2011 and Wiwiek Utami Dewi in 2014 have addressed this issue in the papers related to the lining process of the RX1220 rocket motor. Sanding is the method used to prepare the surface of thermal protector material for the lining process (Dewi, 2014). This paper also describes the change of the epoxy resin type to optimize the lining process but did not provide further discussion on fiber fraction. Jeswani and Roux in 2010 have investigated the correlation of fiber volume fraction and resin viscosity. The paper stated that complete wet out of the dry fiber reinforcement by the liquid resin depends strongly on the fiber volume fraction and the resin viscosity.

Similar research was conducted by Abdurohman et al. in 2018. The author made a comparison between hand lay-up, vacuum bagging, and vacuum infusion towards e-glass epoxy composite. The author found that the hand lay-up method resulted in a 61% fiber mass fraction, while the vacuum bagging resulted in a 73% fiber mass fraction. Recent research to improve laminate quality in wet lay-up/vacuum bagging process by magnet assisted composite manufacturing (MACM) has been conducted by Mehrad Amirkhosravi et al. in 2017. The paper stated that laminate quality successfully improved the fiber volume fraction. The fiber volume fraction increased more than 55% from 17% to 27% and void content decreased by 53% to under 3% compared to the laminates made without magnetic pressure.

Flinn (2007) has investigated the influence of peel ply type on adhesive bonding on composite. The fracture path during removal of peel ply has a strong effect on the quality of the bond. Peel ply remnants on the substrate surface were shown to be detrimental to bond quality. Flinn performed the surface topography analysis by SEM. Studies on the influence of surface treatment type, in the effectiveness of structural adhesive bonding, for carbon fiber reinforced composites have been conducted by Martínez-Landeros et al (2019). The paper correlates the effectiveness of surface preparation type (pre-bonding), such as solvent cleaning, sanding (mechanical abrade), chemical etching (alkaline and acid), and peeling of the sacrificial surface layer (peel ply) of carbon fiber reinforced composite (CFRC) test specimens, with the corresponding effect to the final strength of adhesive bonding. It was performed using several techniques including SEM. Flinn (2005), Kanerva and Saarela (2013), Wegman and Van Twisk (2013) and Holtmannspotter et al. (2013) also conducted similar research.

2.2. Problem Definition

When manufacturing thermal protector materials, fiberglass and fiber carbon are impregnated with epoxy resin. The fiber is laid down one layer at a time and the epoxy resin is poured on and spread with a roller or a squeegee. Multiple layers are often required to achieve the designated thickness. The fraction of fiber reinforcement is very important in determining the mechanical and thermal properties of the composite.

Higher fiber fraction provides better mechanical strength. Fiber mass fraction in typical thermal protector material manufactured by wet lay-up is low (less than 50%). Bleeder cloth then added to adsorbs the excess resin. Bleeder schedule affects the fiber fraction of composites because it provides different adsorption ability.

The surface of the thermal protector material must be fairly rough to provide better bonding with the liner layer in the rocket motor. Sanding and filing are the common method used to prepare the thermal protector material for the lining process. The sanding or filing method has some disadvantages: (1) the surface is too large so it requires more working time, (2) it results in non-uniform roughness since it is manually applied by hand, (3) the abrasive work changes the thickness of the thermal protector material, (4) the excess fiber particulate becomes airborne during sanding. It can get stuck in the lungs. It is better to eliminate the exposure.

The application of bleeder cloth is not only to adsorbs excess resin but also to gives better surface preparation. It increases efficiency since it does not require more surface preparation work. The laminates surface will be fairly rough after bleeder cloth removal. The effect of bleeder cloth on the laminates surface were investigated by preparing laminates with peel ply and perforated release film.

2.3. Method

Thermal protector material samples are made of fiberglass and fiber carbon composite. Epoxy resin is used as the matrix. It is manufactured by wet lay-up and wet vacuum bagging methods. The specifications of the material are shown in Table 2-1.

Table 2-1: Material Specifications

No	Materials	Specifications
1	Epoxy A	Epoxy Bisphenol A. Justus Kimia Raya
2	Hardener EPH 555	Hardener Cycloaliphatic Amine. Justus Kimia Raya.
3	LP3	Liquid Polysulfide LP3. Morton Thiokol USA.
4	Fiber Glass Cloth	E-Glass. Twill Weave. Thickness 0,22 mm ± 10%. Fiber areal weight (FAW) 200 g/m^2. Density 1 gr/cm^3.
5	Fiber Carbon	Plain Weave, Carbon 3K. Density 1,78 g/cm^3. Tensile Strength 3310 MPa. Tensile Modulus 240 GPa. Fiber areal weight (FAW) 220 g/cm^3
6	Peel Ply	Nylon. FAW 85 g/m^2. Justus Kimia Raya
7	Breather Ply	Polyester non-woven. White. FAW 150 g/m^2. Width 1500 mm. Heat resistance up to 200^{o}C. Justus Kimia Raya.
8	Perforated Release Film	Polyethylene with small pores. Thickness 40 micron. Width 1500 mm. Justus Kimia Raya.

In this study, 9 samples of composite material made by different bleeder schedules. Sample 1 was manufactured without bleeder cloth. Sample 2 - 5 were manufactured by hand lay-up. Meanwhile, samples 6 - 9 were manufactured by vacuum bagging. Vacuum bagging was applied to provide better suction for the excess resin to escape. The mold was a working table, topped with 1 cm thick tempered glass. The tempered glass was coated with a Frekote 700-NC release agent. The composite samples were cured at room temperature for 24 hours. The room temperature was 23^{o}C – 25^{o}C with humidity 75% - 78%. Thermal protector material samples are presented in Table 2-2 and the vacuum bagging method is presented in Figure 2-1.

Table 2-2: Thermal Protector Material Samples

Sample Name	Resin Compositions (% wt.)	Bleeder Schedules
Sample 1B	Epoxy A: EPH555 : LP3 = 50% : 25% : 25%	No Bleeder
Sample 2B		Peel Ply Nylon + Fiberglass
Sample 3B		Peel Ply Nylon + Breather Fabric
Sample 4B		Perforated Release Film + Fiberglass
Sample 5B		Perforated Release Film + Breather Fabric
Sample 6B		Peel Ply Nylon + Fiberglass + Vacuum
Sample 7B		Peel Ply Nylon + Breather Fabric + Vacuum
Sample 8B		Perforated Release Film + Fiberglass + Vacuum
Sample 9B		Perforated Release Film + Breather Fabric + Vacuum

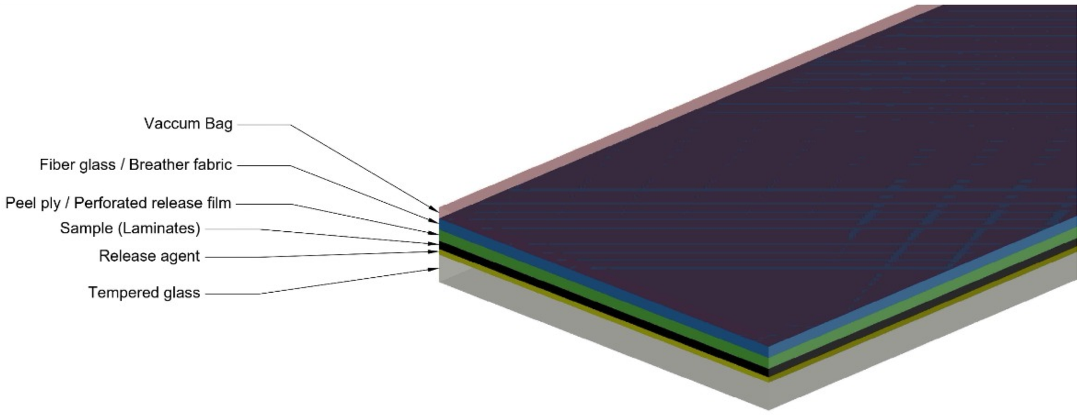


Figure 2-1: Arrangement in Vacuum Bagging Method

The fiber mass fractions were determined by eq. 2-1.

$$X_f = {}^{m_f}/_{m_t} \tag{2-1}$$

and

$$m_f = A_f \times F_{aw} \tag{2-2}$$

According to Table 2-1, the fiber areal weight (F_{aw}) of fiberglass is 200 gr/m^2 and F_{aw} of fiber carbon is 220 gr/m^2.

When the composites cured, the bleeder is removed and the samples were trimmed to provide a uniform area. The total mass of the samples is obtained from weighing the trimmed samples. The topography of the laminate surfaces was analyzed after bleeder removal through scanning electron microscopy (SEM). The SEM was Mini-SEM Phenom World. The viscosity of the epoxy resin was measured by Brookfield DVII+ Pro viscometer.

3. Result and Analysis

The viscosity of the epoxy resin is 451 – 472 cp. The fiber mass fraction of the thermal protector samples are shown in Tables 3-1 below.

Table 3-1: Fiber Fraction of Thermal Protector Samples

Sample Name	Bleeder Schedules	Fiber Mass Fraction
Sample 1B	No Bleeder	56.27%
Sample 2B	Peel Ply Nylon + Fiberglass	56.14%
Sample 3B	Peel Ply Nylon + Breather Fabric	56.78%
Sample 4B	Perforated Release Film + Fiberglass	47.72%
Sample 5B	Perforated Release Film + Breather Fabric	49.18%
Sample 6B	Peel Ply Nylon + Fiberglass + Vacuum	56.59%
Sample 7B	Peel Ply Nylon + Breather Fabric + Vacuum	64.94%
Sample 8B	Perforated Release Film + Fiberglass + Vacuum	58.16%
Sample 9B	Perforated Release Film + Breather Fabric + Vacuum	66.43%

According to Table 3-1, the fiber mass fraction of the thermal protector samples without bleeder has already surpassed 56%. The addition of bleeder cloth did not affect the fiber mass fraction in the hand lay-up method. When peel ply was replaced by perforated release film, the fiber mass fraction decrease below 50% (Figure 3-1). The perforated release film is made of plastic therefore it did not adsorb the excess resin well enough compare to peel ply. Perforated release film allowed the fiber to adsorb more resin therefore the fiber mass fraction decreased. Nevertheless, breather fabric provided better adsorption as a second layer of bleeder when perforated release film was the first layer of the bleeder (sample 5B). The adsorption increase 1.46% from 47.72% to 49.18%. In opposite, neither fiberglass nor breather fabric increased the fiber mass fraction when peel ply was used as the first layer of the bleeder. When peel ply was used as the first layer, the excess resin has already been adsorbed in the peel ply thus the fiberglass and breather fabric cannot absorb more. Fiberglass was not a decent bleeder in the hand lay-up method. It poorly adsorbed the excess resin hence it decreased the fiber mass fraction.

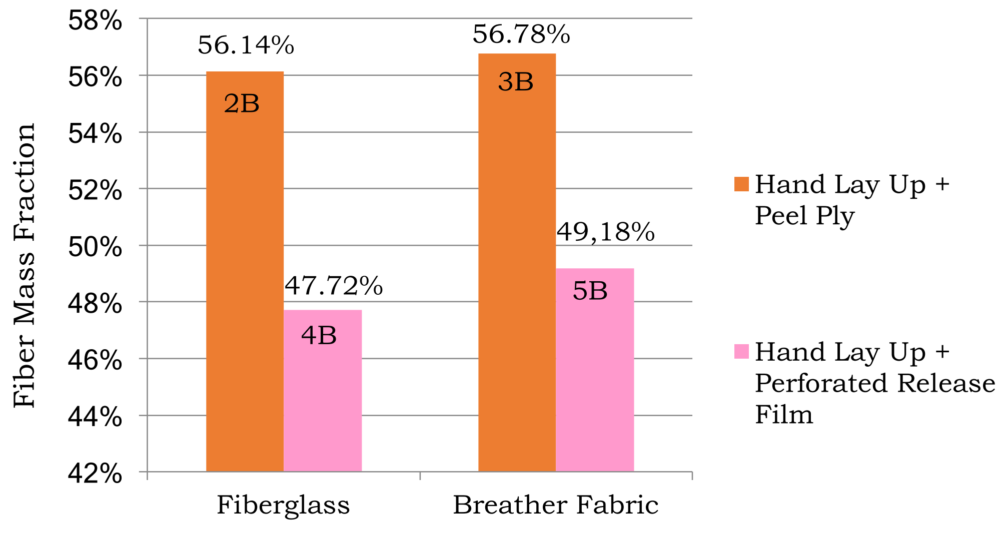


Figure 3-1: Fiber Mass Fraction Comparison on Hand Lay-up Manufacturing Process

When the vacuum was applied to sample 6 – 9, the fiber mass fraction increased significantly (Figure 3-2) from 56% to 66%. Whether the vacuum bagging method used peel ply or perforated release film, the fiber mass fraction increased. The interesting finding is that the perforated release film provided a higher fiber mass fraction than the peel ply. It is the opposite of the hand lay-up method. The pores throughout the film surface facilitated better excess resin transfer to the second layer bleeder (fiberglass and breather fabric). Similar to the hand lay-up method, fiberglass was not a decent bleeder in the vacuum bagging method. Accordingly, it confirmed the fiberglass's poor adsorption ability as a bleeder. Breather fabric showed great performance in the vacuum bagging method.

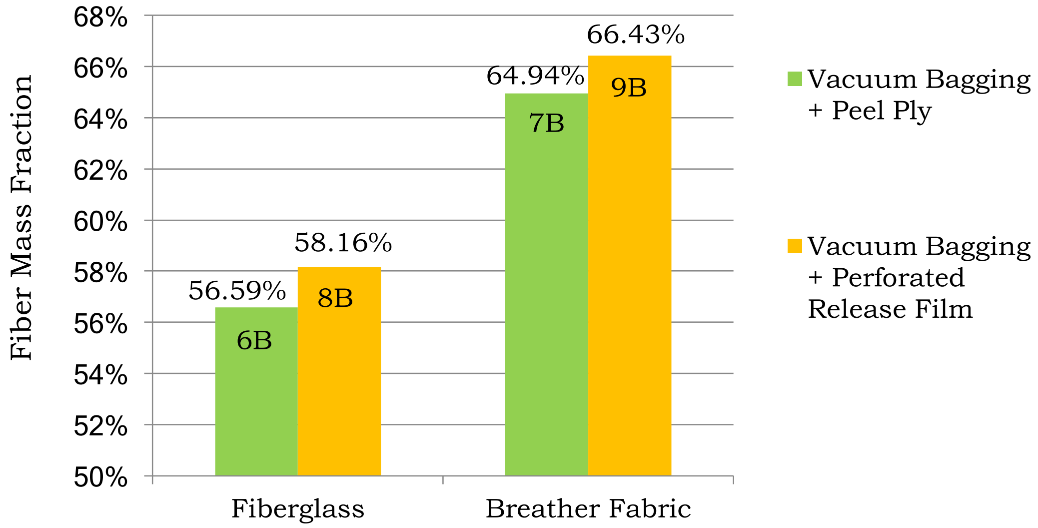


Figure 3-2: Fiber Mass Fraction Comparison on Vacuum Bagging Manufacturing Process

The composite surface topography was the author’s concern because this work aims to eliminate sanding or filing as the surface preparation method for the lining process. The scanning electron microscope (SEM) analysis was performed after the removal of peel ply and perforated release film. Representative SEM images of the composite surface are shown in Figures 3-3 to 3-7.

Figure 3-3 is the surface of the thermal protector material without bleeder. This surface is different from any other surface. It has rich resin but the fiber pattern is seen. In some areas, fiber is not covered thoroughly with resin (red arrows). The yarn remnants were found imbedded in the surface (blue arrows). They were suspected to be left-out brush yarn or chopped fibers. EDS analysis should be performed to determine the source.

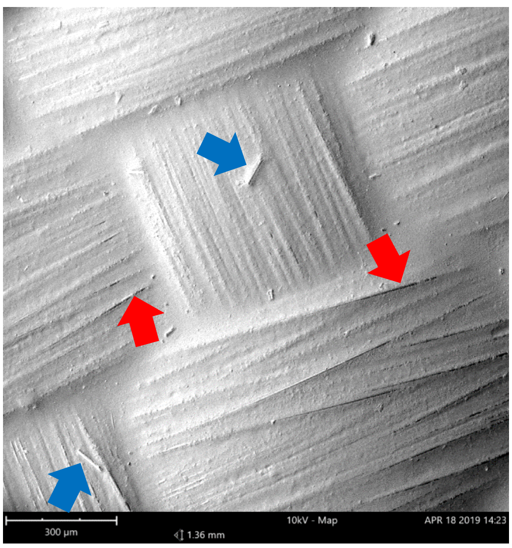
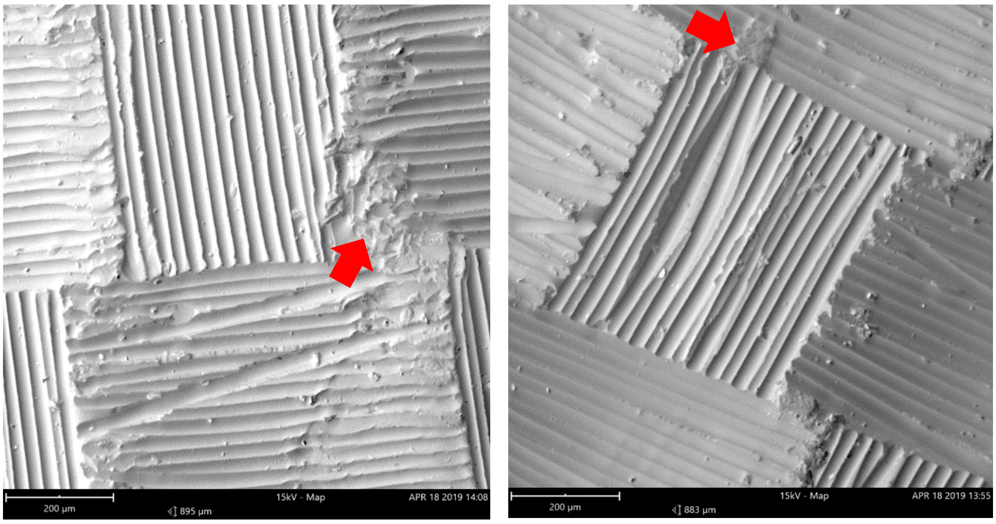


Figure 3-3: SEM Image of Composite’s Surface Without Bleeder Cloth. The red arrows are fibers were not covered by resin, and the blue arrows are the yarn remnants that were embedded in the surface

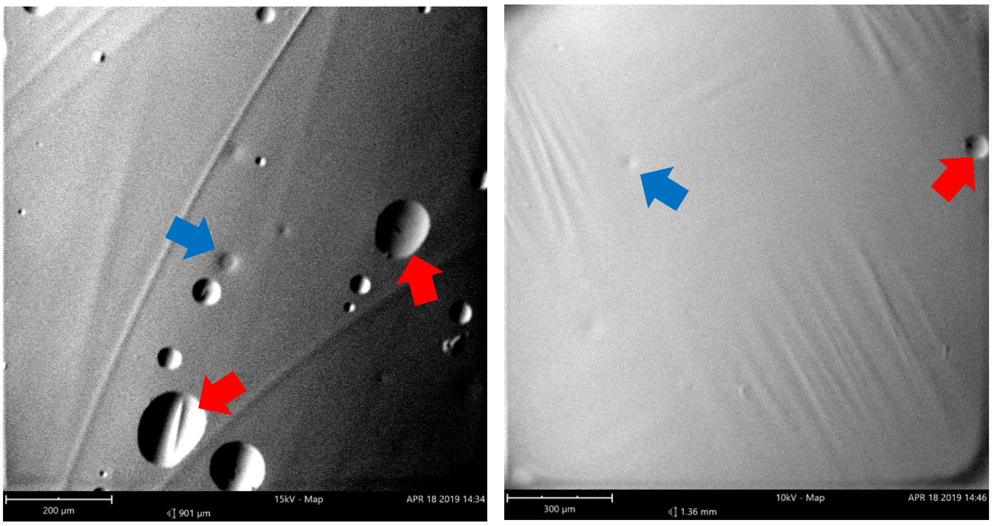


(a) 2B (b) 3B

Figure 3-4: Composite with Bleeder Schedule: (a) Sample 2B - Peel Ply & Fiberglass, and (b) Sample 3B - Peel Ply & Breather Fabric. The red arrows are ductile wisps on the epoxy surface.

Figure 3-4 is the comparison of the surface after the removal of peel ply - fiberglass (2B) and peel ply - breather fabric (3B). Both were manufactured by hand lay-up method. The imprints of the nylon peel ply weave are seen on the surface. The surface is fairly rough. Ductile wisps on the epoxy surface are also visible (red arrows). These are typical of a ductile polymer fracture (Flinn, 2007). 2B has more ductile wisps than 3B. Since tendrils are not present on the surface, it is likely that no remnants of the nylon peel ply fibers.

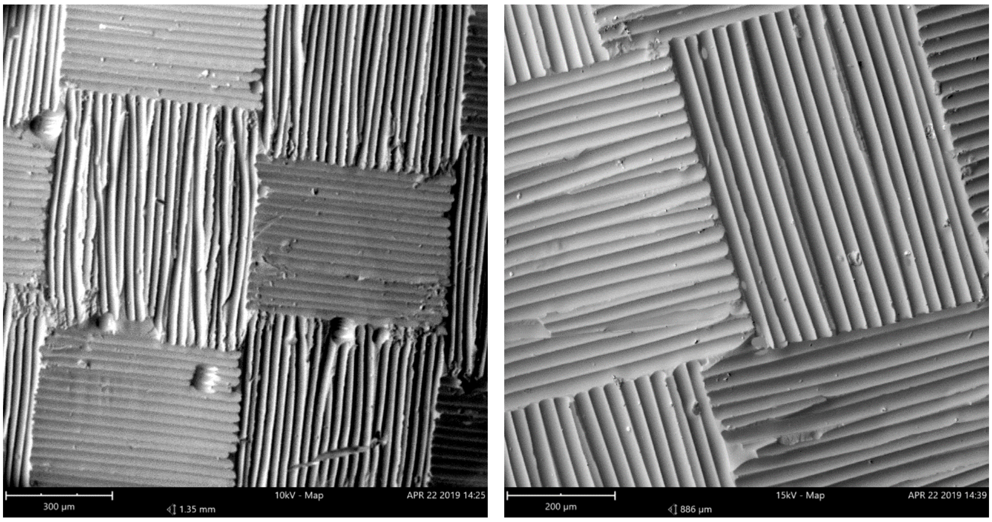
Figure 3-5 is the comparison of the surface after the removal of perforated release film - fiberglass (4B) and perforated release film - breather fabric (5B). They both were manufactured by hand lay-up method. There are no imprints seen on the surface. The surface is smooth with several voids. Several voids are likely air trapped below the perforated release film (red arrows). The imprints of the pores from perforated release film are seen as well on the surface (blue arrows). Also, the imprints of the plastics folding from uneven hand lay-up work are shown on the surface.



(a) 4B (b) 5B

Figure 3-5: Composite with Bleeder Schedule: (a) Sample 4B – Perforated Release Film & Fiberglass, and (b) Sample 5B – Perforated Release Film & Breather Fabric. The red arrows are imprints of the pores from the perforated release film, and the blue arrows are voids made from air trapped below the perforated release film.

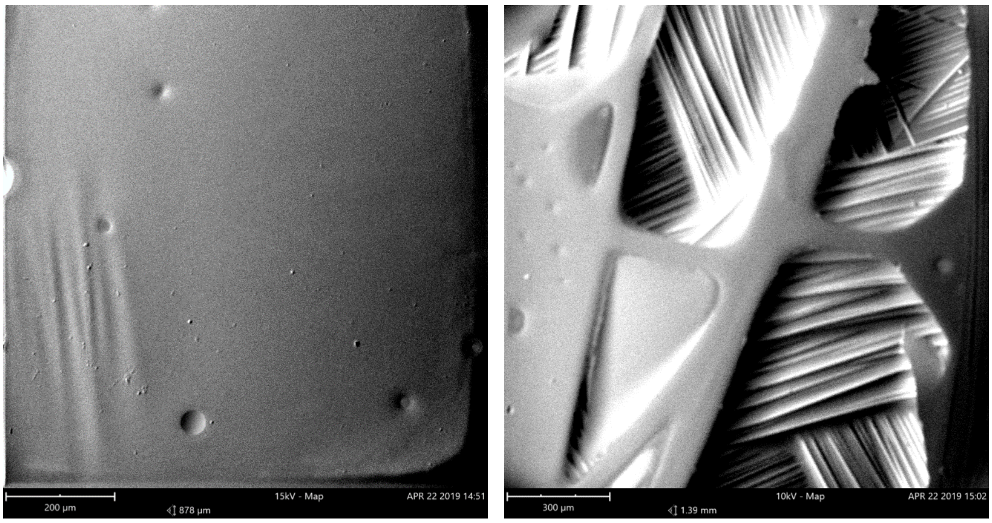
Figure 3-6 is the comparison of the surface after the removal of peel ply - fiberglass (6B) and peel ply - breather fabric (7B) manufactured by vacuum bagging. The imprints of the nylon peel ply weave are visible on the surface. Few ductile polymer fractures are visible but not too large compare to Figures 3-4. Vacuum bagging reduces the existence of the ductile wisps. Figure 3-6 shows that 6B has more ductile polymer fractures than 7B. This indicates that breather fabric can adsorb more resin than fiberglass. It is shown in a higher fiber mass fraction of 7B than 6B. There are no remnants of the nylon peel ply fibers on both surfaces since tendrils are not visible.



(a) 6B (b) 7B

Figure 3-6: Composite with Bleeder Schedule: (a) Sample 6B - Peel Ply & Fiberglass, and (b) Sample 7B - Peel Ply & Breather Fabric. Both were manufactured by vacuum bagging.

Figure 3-7 is the comparison of the surface after the removal of perforated release film - fiberglass (8B) and perforated release film - breather fabric (9B) manufactured by vacuum bagging. Similar to Figures 3-5, there are no imprints seen on the surface. Several voids are visible on 8B but there were not many compare to the previous one (Figure 3-5). Vacuum bagging reduces the existence of trapped air. The surface of the 9B is surprising. There are large voids with fibers on their bottom. During the vacuum condition, the resin was drawn out from the fiber's surface unevenly. It created those large voids.



(a) 8B (b) 9B

Figure 3-7: Composite with Bleeder Schedule: (a) Sample 8B – Perforated Release Film & Fiberglass, and (b) Sample 9B – Perforated Release Film & Breather Fabric. Both were manufactured by vacuum bagging.

The ductile wisps and the tendril on the composite surface require further analysis by EDS (Energy Dispersive Spectrometry) or XPS (X-ray Photoelectron Spectroscopy) to confirm the source. Nylon peel plies usually coated with a release agent such as silicone, it is important to ensure that the silicone release agent is not transferred to the laminate's surface. The silicone will contaminate the surface, therefore, decreasing the bonding strength (Hart-Smith et al, 1996). EDS or XPS analysis should be performed to determine whether the silicone release agent is transferred to the laminates.

4. Conclusions

The highest fiber mass fraction (66.49%) was achieved by vacuum bagging manufacturing process with perforated release film and breather fabric bleeder schedule. Meanwhile, the lowest fiber fraction (47.72%) was achieved by hand lay-up process with peel ply and fiberglass bleeder schedule. The fiber mass fraction of thermal protector material manufactured by hand lay-up can reach a maximum of 56.78%, whereas vacuum bagging can reach a maximum of 66.43%. In terms of fiber mass fraction, peel ply and breather fabric combination are the best bleeder schedule for the hand lay-up method meanwhile perforated release film and breather fabric are the best bleeders for the vacuum bagging method. In both methods, breather fabric adsorbs more excess resin than fiberglass. Composite surface topography obtained from peel ply is visible on the surface. Meanwhile, the surface topography obtained from the perforated release film is not visible. This paper recommends peel ply usage in the thermal protector manufacturing process to replace the sanding or filling method that the author use nowadays.

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Contributorship Statement

WUD is the main contributor. Both WUD and RS did the experiment and analysis.

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