Study of Permeability Testing Methods for Composite Laminates

A Senior Project

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Bachelor of Science, Materials Engineering

by

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ABSTRACT

Permeability measurements are strong indicators of the effectiveness of evacuation channels within a composite prepreg. The effectiveness of these evacuation channels directly relates to the void content within a processed composite laminate. The goal of Toray Advanced Composites was to compare two permeability tests and determine which one was more reliable, accurate, and economical. The first permeability test method was the Cirrus method, designed by Cirrus Aircraft, a customer of Toray Advanced Composites. The second test was ASTM D8132, the standard method for permeability testing. Additionally, Toray sought to investigate the effect out-time had on the permeability of three different prepreg products using the more feasible permeability test option. For the comparison stage of the project, both the ASTM and Cirrus tests were run 5 times. Different modifications and techniques were recorded during these tests for future consideration on how to improve test efficiency. The Cirrus data yielded an average permeability value of 3.98 x 10^{-14} m^2; while the ASTM test yielded an average permeability value of 7.4 x 10^{-12} m^2. Both tests yielded repeatable data. The Cirrus data yielded a standard deviation of 1.5 x 10^{-14} m^2, and the ASTM data had a standard deviation of 1.8 x 10^{-12} m^2. The order of magnitude difference between the two data sets was determined to be the result of differing sample preparation methods for each test. Qualitative analysis was also used to determine which test was more feasible based on ease of setup, total run time, cost, and amount of material used per test. The ASTM test, on average, took two hours less to prepare and conduct the test compared to the Cirrus test and used 45 in^2 less of material per sample. It also cost around $3550 less to prepare and run the test. Due to the minimal resources required to run the ASTM test and its certification as a standard test method, the ASTM test was determined to be the more efficient and more feasible option. The modifications and techniques to run each test more efficiently were written into standard operating procedures for future use and development by Toray engineers and technicians. However, due to the COVID-19 outbreak the out-time experiments were cancelled, and all further work was ended.

Keywords: Materials Engineering, composites, prepreg, laminate, out-of-autoclave, vacuum-bag only, epoxy, carbon fiber, curing, void, out-time, permeability, ASTM D8132

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We would also like to thank Dr. Mohsen Kivy for his advisement in the theoretical understanding of the transport phenomena involved in this project. Lastly, we would like the thank Dr. Blair London of the Cal Poly Materials Engineering Department for organizing this project and providing us unabridged guidance throughout the entire project despite the pandemic. His dedication to his students is unmatched and has taught us the values of perseverance and commitment to excellence no matter the circumstance, and his leadership navigated us through these times.

INTRODUCTION

I. BACKGROUND

COMPOSITE PREPREGS

Composites are fiber-reinforced matrix materials with distinct phases which have unique properties not exhibited by either component alone. Prepregs lay the foundation for composite manufacturing. At this point the fiber architecture is set which dictates fiber and matrix properties. Composite prepregs are fibers pre-impregnated with catalyzed but partially cured resin. They are produced by saturating a fiber reinforcement with a liquid thermoset resin. The excess resin is removed, and the remaining resin undergoes a partial cure. Here the thermoset transforms from a liquid to a pliable solid state known as "B-stage." B-stage prepregs are stored in freezers until used to prevent premature curing. The curing process is completed with the application of pressure and heat [1].

MANUFACTURING METHODS

A combination of external pressure, vacuum, and heat is applied to an assembly of composite layers to consolidate and densify separate plies of prepreg into a solid laminate. High cure temperatures and pressures are required to initiate and complete the curing process. High pressures provide the force needed for the highly viscous resin to flow in the mold and consolidate the prepregs [2]. Traditionally this is accomplished with an autoclave, a high-pressure furnace that has the ability to produce high quality products. However, autoclave processing has high capital costs, space constraints and high energy consumption. These financial restraints along with government regulations to minimize energy consumption resulted in a trend towards out-of-autoclave processing methods for composite laminates [3]. In general, out-of-autoclave processing applies less pressure at a lower temperature than the autoclave to reduce equipment costs and maintenance [3]. Vacuum-bag only (VBO) is an out-of-autoclave process done in a convection oven. VBO parts have a slightly higher content of voids, which are defects within the laminate, compared to autoclaved parts. Voids are entrapped air, moisture, or cure-generated volatiles within the matrix. The presence of these defects affects the mechanical properties and by extension, the performance of the composite.

MECHANICAL EFFECT OF VOIDS

Voids within the laminate diminish the mechanical properties of the composite. In a study conducted by Fox and Agius, two laminate samples were prepared, one with 0.3 vol% void content and one with 1.5 vol% void content (Figure 1). There was a 21% decrease in fracture toughness between the 0.3 vol% void content and 1.5 vol% void content laminate, suggesting that as void content increases fracture toughness decreases [4].

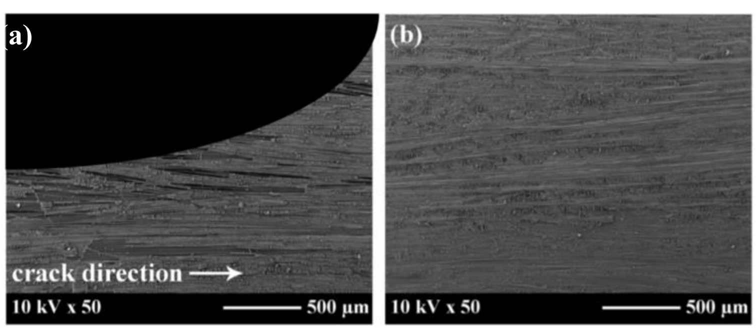


Figure 1. a) SEM image shows a higher void content laminate with microcracks as a result of a lower fracture toughness. b) This sample has a lower void content and higher fracture toughness with no microcracks [4].

In a separate study conducted by Liu and Zhang, tensile, shear and flexural strength along with flexural modulus were tested with respect to increasing void content, ranging from 0.6 vol% to 3.2 vol% (Figure 2). For tensile strength, there was 14% decrease in tensile strength going from 0.6% to 3.2% void content. Shear strength and flexural strength had similar trends, with an 18% and 22% decrease, respectively, between the lower void content laminate and the higher void content laminate. Flexural modulus was also examined but is not depicted in Figure 2. For flexural modulus there was a 18% decrease in the modulus going from low to high void content [5].

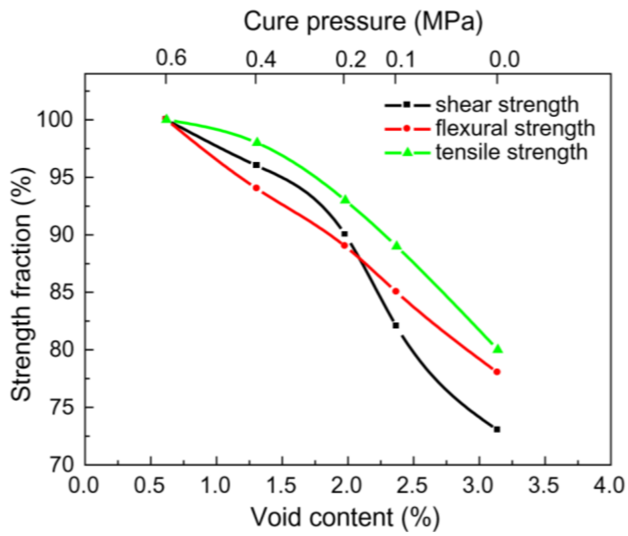


Figure 2. Overall strength decreases with increasing void content [5].

Void Formation and Prevention

Voids are a result of improper resin flow. Resin flow is a function of viscosity, a measure of a fluid's resistance to shear stress. The viscosity of the resin is higher within the tows of fiber than in the inter-tow spaces due to the shear response in the fiber-resin interface. This results in dry cores within the tows, surrounded by resin (Figure 3a). Proper resin viscosity leads to full impregnation throughout the laminate regardless of the capillary action exhibited by the resin. However, if the viscosity is too low, the resin will pass over the tow. On the other hand, if the viscosity is too high, the resin is unable to fully infiltrate the tow (Figure 3b). Both situations lead to void formation within the laminate [6].

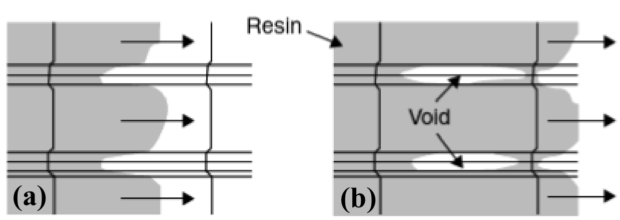


Figure 3. (a) Capillary action forms void within the tows (b) Void formation within tows from improper flow [3].

Air evacuation is a mechanism implemented into laminates to minimize void content once the final cure is complete. Air evacuation is the unidirectional flow of compressed air through a permeable and porous membrane. In typical autoclave processing, the high pressure from the applied vacuum and high heat allow for easy air evacuation in the composite laminate. However, in VBO processing, the vacuum pressure and heat are significantly less. Therefore, out-of-autoclave prepregs are characterized by the partial impregnation of fibers where dry fibers or channels within the prepreg tows are implemented to aid in proper air evacuation. These channels, known as EVaCs or engineering vacuum channels, remain open during the initial stage of curing for air to evacuate (Figure 4) [1]. This produces VBO laminates with low void contents comparable to autoclave laminates [7].

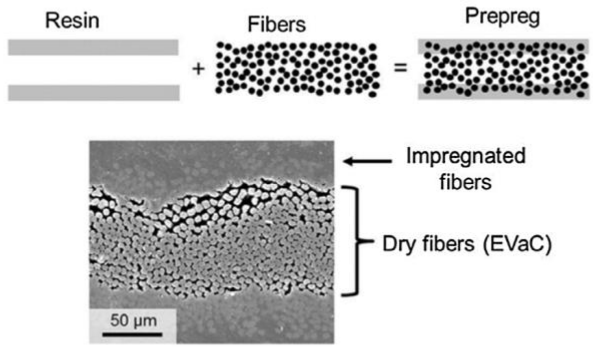


Figure 4. Prepreg consists of partially cured resin and fibers with an evacuation channel of dry fibers [1].

In addition, the formation of voids is a function of the initial degree of cure. The degree of cure is monitored by out-time. Out-time is a hold time where the prepregs are exposed to ambient temperatures before the cure goes to completion. The epoxy resin in the prepregs undergo chemical changes which affect processability and final part quality [8]. As the resin cures during out-time, a change in viscosity occurs which can lead to the formation of voids. A non-ideal viscosity of resin impedes the full impregnation of the tows, leading to an increased number of voids in the VBO laminate (Figure 5). Therefore, it is important to understand how viscosity changes as function of out-time [9].

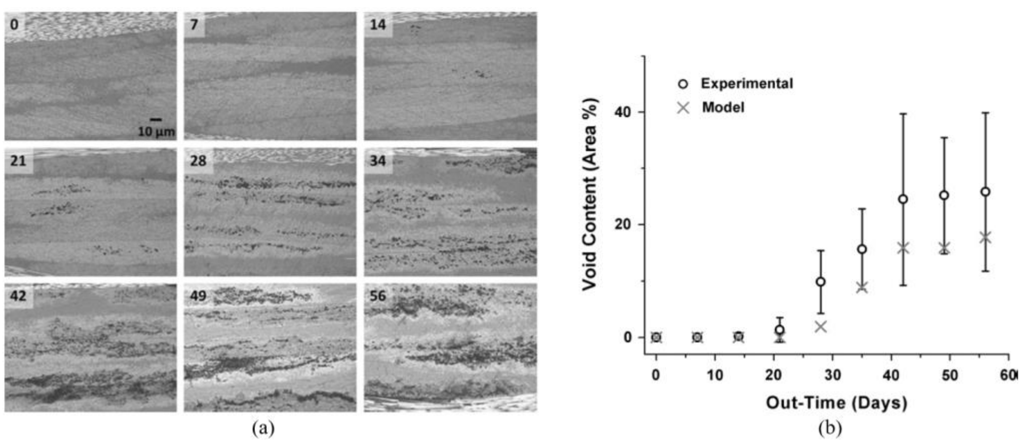


Figure 5. (a) SEM images of tow impregnation in samples as a function of out-time (days) (b) Corresponding data demonstrating sinusoidal relationship between out-time and void content [8].

PERMEABILITY

Permeability is defined as the momentum transport of a fluid through a porous medium [10]. For the context of this project, permeability is the measure of air flow through the in-plane direction of the composite laminate. Voids are the pores within the laminate. Subsequently, void content within the laminate can be monitored through permeability measurements because porosity and permeability are directly related. Permeability is modeled mathematically based on Darcy flow and the ideal gas law [7]. The ideal gas law is $PV = nRT$, where P is pressure; V is volume; n is moles of fluid; R is the gas constant, and T is temperature. Equation 1 is the derivative of the ideal gas law where Q is mass flow or volumetric flow rate (V/t) and $\dot{n}$ is the molar flow rate of air.

$$PQ = \dot{n}RT \qquad \text{Equation 1.}$$

Darcy's law, Equation 2, relates permeability to the ideal gas law. Here, K is permeability, μ is dynamic viscosity and A is the cross-sectional area of the laminate [10].

$$Q = -\frac{KA}{\mu} \cdot \frac{dP}{dx} \qquad \text{Equation 2.}$$

By combining Equations 1 and 2, permeability can be calculated through various controlled experiments that measure different variables to monitor the void content in laminates.

II. TORAY ADVANCED COMPOSITES | MORGAN HILL, CA

Toray Advanced Composites supplies polymer matrix composite materials to a wide range of aerospace fields, including general aviation, military aircraft, and space exploration. These materials provide lightweight yet strong structures critical to the success in its application. Additionally, Toray is the leading global supplier in thermoset prepregs for out-of-autoclave/vacuum-bag-only applications, the lower cost processing option when compared to traditional autoclave processing [11]. Toray aims to supply their customers with the highest quality product and continues to push the industry standard.

III. PROBLEM STATEMENT

Permeability measures the void content in composite laminates which affects the mechanical properties of the final product. Toray Advanced Composites, located in Morgan Hill, CA, would like to provide their customers with information regarding the permeability of their laminates. Cirrus Aircraft, a customer of Toray, developed a method of measuring the permeability. One of the goals of this project is to provide a qualitative analysis on the reliability of this test method compared to ASTM D8132, the standard test method for determination of prepreg impregnation by permeability measurement. The second goal of this project was to determine how out-time, the total time the prepreg is exposed to ambient temperatures, affects permeability. The Toray prepregs to be tested were HTS45 mated with TC275 resin, HTS40 mated with TC275-1 resin and 1062E mated with TC380 resin. However, due to the COVID-19 outbreak this part of the project was not completed.

EXPERIMENTAL PROCEDURE

I. SAFETY

Standard lab safety procedures were followed for the duration of experimental testing. Proper lab attire included long pants, closed-toed shoes, and safety glasses. Furthermore, nitrile gloves were used to prevent skin irritation and small cuts from handling of the carbon fiber prepreg samples. Experimental testing was done in a safe laboratory setting with a partner. If a problem did arise, the appropriate parties were contacted including the lab technician and advisor to help alleviate the situation.

II. CIRRUS TEST PROCEDURE

Sample Preparation

Four plies of TC-275 prepreg measuring 3.38" x 3.35" were cut using Kevlar shears. The TC-275 is an epoxy matrix, carbon fiber composite prepreg system. The four plies were laid up on top of each other oriented in the 0° direction. The 4-ply lay-up was debulked for 1 hour at -25 inHg using a vacuum bag apparatus. The debulking set up involves layers to aid in the release of the laminate from the table including breather material, FEP, and TX-040 films which all exceeded the dimensions of the prepreg lay-up. The layers of debulking were as follows from top (vacuum bag) to bottom (table): breather material, FEP, TX-040, prepreg lay-up, FEP. In the absence of a debulking table, a pseudo-debulking apparatus was constructed using chromate tape lining, vacuum bag material, and a vacuum port. After the debulk, the width, height, and thickness of the sample were recorded.

Permeability Testing

After debulking the 4-ply laminate, the sample was placed in the Cirrus testing apparatus as shown in Figures 6 and 7. Vacuum was applied on both sides of the apparatus for one hour using two Quick Connect valves. A leak-free apparatus is essential to proper testing. This is ensured by testing that the vacuum pressure is maintained after applying vacuum for a short period of time. After a one-hour vacuum hold to establish pressure, one Quick Connect valve was removed and replaced with a COHO mass flow meter. The COHO measured the air flow as it traveled from one side of the apparatus through the cross section of the laminate to the other side of the apparatus. The mass flow reading (mL/minute) was recorded every minute for ten minutes.

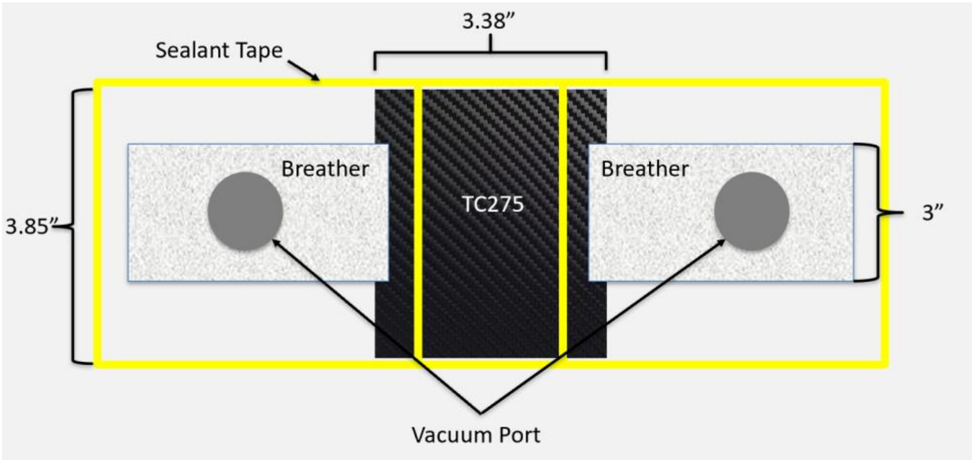


Figure 6. Model of the Cirrus test apparatus, complete with the sealant tape outline, breather cloth, and vacuum ports.

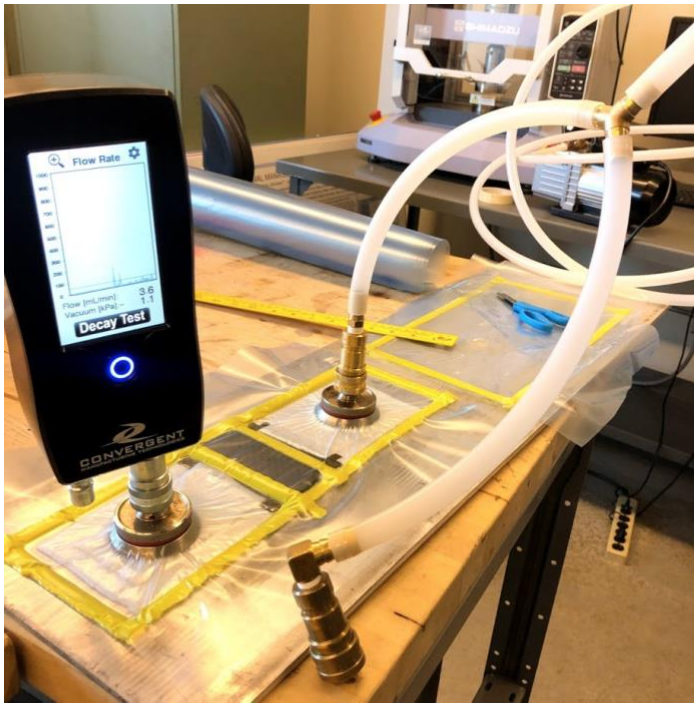


Figure 7. Cirrus apparatus with the COHO mass flow meter attached to the left side vacuum port.

Calculations

The mass flow measurements, *Q*, were averaged, converted to m^2, and inputted into Equation 3 to solve for permeability, *K*. Equation 3 is a derivative of the Ideal Gas Law and Darcy's Law (Equations 1 and 2) given by Cirrus Aircraft engineers.

$$K = \frac{2Q\mu L}{A}\left(\frac{Po}{Po^2 - PL^2}\right) \qquad \text{Equation 3.}$$

Where:

K is the permeability (m^2)
Q is the average calculated volumetric flow rate (m^3/s)
μ is the dynamic viscosity of air (Pa*s)
L is the length of the laminate (m)
A is the cross-sectional area of the laminate (m^2)
P_0 is the absolute pressure of the atmosphere (Pa)
P_L is the absolute pressure of the vacuum (Pa)

III. ASTM TEST PROCEDURE [12]

Sample Preparation

A 1" x 3" strip of TC-275 prepreg was cut from the stock roll using Kevlar shears. The width and thickness of each strip was recorded. The strip was mounted between two acrylic blocks using a two-part epoxy adhesive. The acrylic blocks measured 1" x 1" x 3" and were cut from stock material. Up to 3 samples were prepared at a time using the 10 grams of two-part epoxy prescribed on the ASTM document. Once the epoxy was applied to the blocks the prepreg strip was sandwiched in between the blocks. It was important to make sure no gaps existed between the acrylic and the epoxy as this would lead to leaking air during testing. Any excess epoxy was cleaned off and the sample was left to dry for one hour on a plastic cup.

Permeability Test

The ASTM apparatus, supplied by Toray Advanced Composites, was calibrated for 30 minutes to ensure no leaks were present. The original design needed to be modified slightly by adding sealant tape to the base of the cylinder to further prevent leaks (Figures 8-9). The ends of the prepreg strip in the acrylic mount were cut and the new length was measured. The mounted sample was attached to the top of the cylinder using sealant tape and vacuum was applied for one minute to reach a stabilized pressure within the vessel. After one minute, the vacuum valve was closed and the drop in pressure (inHG) was recorded every minute for 10 minutes. The drop in pressure was due to air leaving the vessel through the cross section of the mounted prepreg strip.

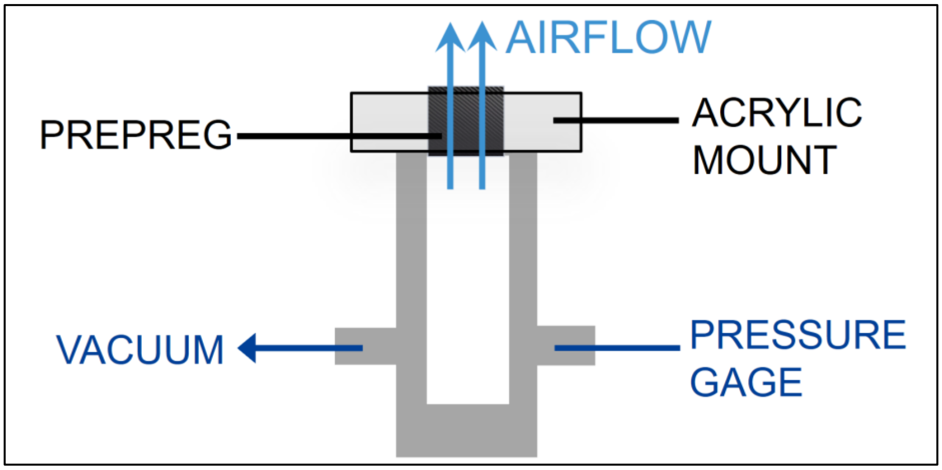


Figure 8. Model of the ASTM test, where the air flow out of the cylinder vessel and through the cross section of the prepreg is denoted by the light blue arrows.

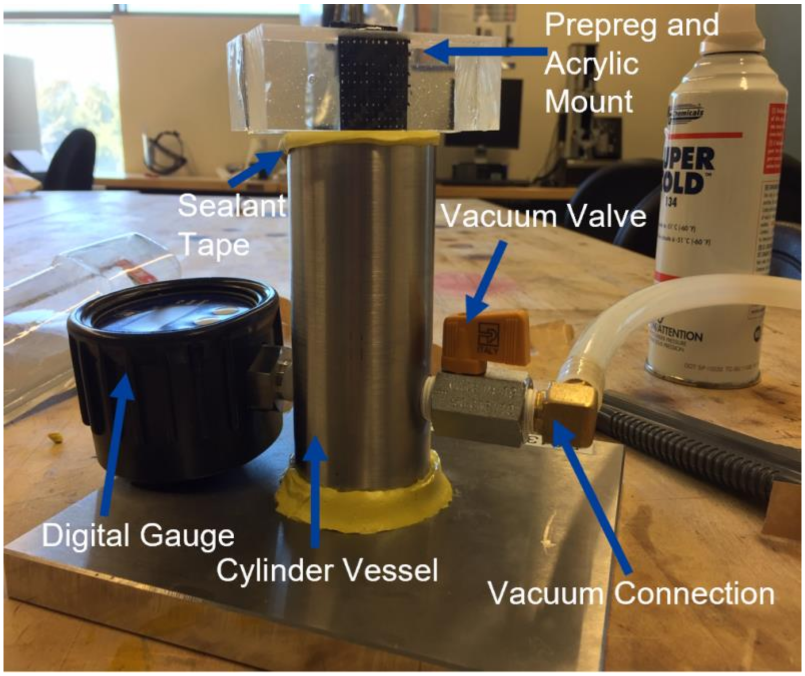


Figure 9. ASTM test apparatus with a mounted sample attached to the cylinder vessel.

Calculations

In order to calculate permeability, Equation 4 was given by the ASTM document. This equation is a derivative of the Ideal Gas Law (Equation 1) and Darcy's Law (Equation 2) specified for a constant volume apparatus. To ease calculations on a spreadsheet, Equation 4 was rearranged to Equation 5, which is written in slope-intercept form (y=mx). The permeability gradient value labeled in Equation 5 is calculated using the measured variable, pressure (*P*), as a function of the atmospheric pressure and initial pressure. This value is graphed as a function of time, and the subsequent the slope of the regression curve is the calculated permeability value, *K*.

$$\ln\left[\frac{(P_{atm}+P)(P_{atm}-P_i)}{(P_{atm}-P)(P_{atm}+P_i)}\right]\frac{KWHP_{atm}}{LV\mu}t \qquad \text{Equation 4. [12]}$$

↓

$$\ln\left[\frac{(P_{atm}+P)(P_{atm}-P_i)}{(P_{atm}-P)(P_{atm}+P_i)}\right]\frac{LV\mu}{WHP_{atm}} = Kt \qquad \text{Equation 5.}$$

Permeability Gradient Value — Constant Values

Where:

P_{atm} is atmospheric pressure (Pa)
P_i is initial pressure (absolute) in the vacuum chamber (Pa)
P is pressure (absolute) in vacuum chamber as a function of time (Pa)
K is the permeability (cm^2)
W is the width of the prepreg sample (cm)
H is the thickness of the prepreg sample (cm)
L is the length of the prepreg sample (cm)
V is the total volume of the vacuum chamber apparatus (cm^3)
μ is the viscosity of air (Pa-s)
t is the time

IV. OUT-TIME EXPERIMENTAL PROCEDURE

Due to the COVID-19 outbreak, this portion of the project was not completed. It was planned for Spring 2020, but the Cal Poly campus was closed for the entire quarter. The out-time experiments were going to use the ASTM permeability test method to measure permeability as a function of out-time over a period of 30 days. The materials to be used were the TC-275 (epoxy/carbon fiber system), TC-275-1E (epoxy/carbon fiber system), and the TC-380 (epoxy/glass fiber system). The samples were to be cut and stored at ambient temperatures. Permeability was to be measured every two days up until day 8 of the experiment. From days 8-14, permeability measurements were to be taken every three days. Finally, from days 14-30 measurements were to be taken every five days. The increase in time intervals between measurements was suggested by Toray Advanced Composites. They believed the permeability value would not change significantly after day 8 based on the recorded maximum out-time of their products which indicates how long a sample can be kept at ambient temperatures before properties would deteriorate.

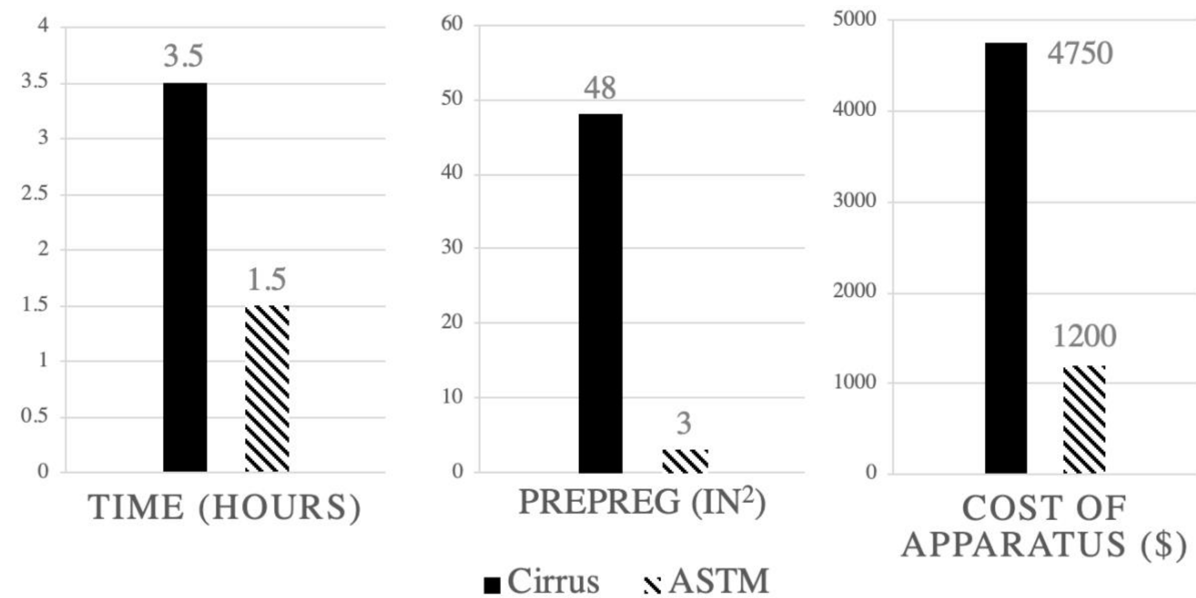
RESULTS

To determine which test yielded more repeatable and accurate data, the Cirrus and the ASTM tests were both run five times. Their calculated permeability values are listed in Table I.

Table I. Permeability Values for Cirrus and ASTM Tests

Test	Cirrus (m^2)	ASTM (m^2)
1	5.55868×10^{-14}	4.62797×10^{-12}
2	3.78451×10^{-14}	7.23505×10^{-12}
3	4.07580×10^{-14}	7.07972×10^{-12}
4	1.99722×10^{-14}	9.15348×10^{-12}
5	4.48253×10^{-14}	8.88612×10^{-12}
Average	**3.98×10^{-14}**	**7.40×10^{-12}**

Additionally, to holistically compare the two test methods, other factors besides permeability were considered. Resources consumed by each test method such as average time for completion and average material used were recorded (Figure 10). In addition, any modifications or techniques that were found to improve the experimental procedure of both tests were recorded in the Toray standard operating procedure documents (Appendix A and B). In this way, ease of operation was also considered.



a) b) (c)

Figure 10. The average resources required for each test including a) time to prepare and run a test on a single sample and b) the amount of prepreg used per test and c) the cost of the apparatus was lower for the ASTM method than the Cirrus test.

DISCUSSION

The Cirrus data had a standard deviation of 1.5 x 10^{-14} while the ASTM data had a standard deviation of 1.8 x 10^{-12}. This indicates low variability within each test method. The concern with the data is that the permeability values between each test differ by two orders of magnitude (Figure 12). The Cirrus results have an order of magnitude of 10^{-14} while the ASTM results have an order of magnitude of 10^{-12}.

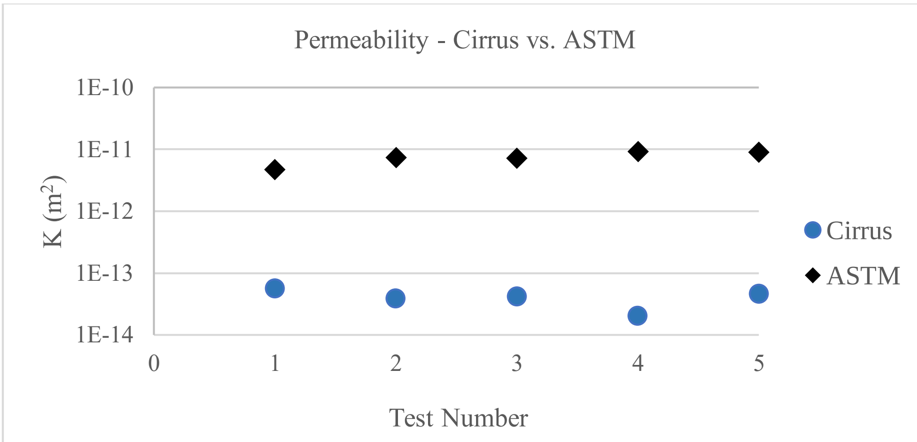


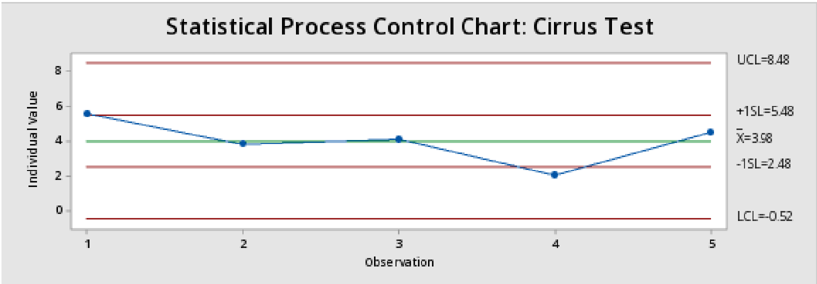
Figure 11. 10^2 difference in magnitude between the Cirrus and ASTM permeability values.

The Cirrus data was within the same order of magnitude as the data collected by Cirrus Aircraft engineer Tyler Tedlund using the same method. The order of magnitude of 10^{-12} was the same and both data sets are close in relative value [13]. No literature data for the ASTM permeability test was found in research. The hypothesis was that the difference in permeability values stems from the difference in the sample preparations for each test. Specifically, the Cirrus test used a 4-ply debulked laminate while the ASTM test used a 1-ply strip of non-debulked prepreg. There were plans to address this difference by running each test again and switch sample preparation procedures: the Cirrus test using a single ply, non-debulked strip, and the ASTM test using a 4-ply, debulked laminate. However, these tests were never conducted due to the COVID-19 outbreak.

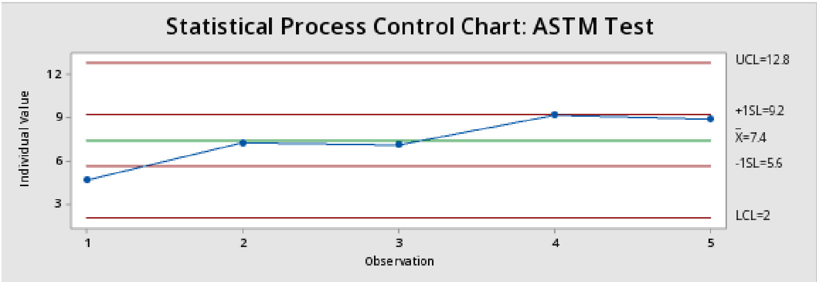
Furthermore, statistical control tests conducted on the data sets determined both of them to be under control which speaks to the quality of the testing method (Figure 11). Statistical control was determined given that the data did not fail any of the following test conditions:

1. 1 point > 3 standard deviations from the center line.
2. 9 points in a row on the same side of the center line.
3. 6 points in a row, all increasing or all decreasing.
4. 14 points in a row, alternating up and down.
5. 2 out of 3 points > 2 standard deviation from the center line on the same side.
6. 4 out of 5 points > 1 standard deviation from the center line on the same side.
7. 15 points in a row within 1 standard deviation on of center line on either side.
8. 8 points in a row > 1 standard deviation from the center line on either side.

Given that only 5 tests were conducted due to the COVID-19 outbreak, statistical control should be reestablished if Toray uses either test method moving forward.



(a)



(b)

Figure 12. Statistical Process Control Charts of (a) Cirrus tests (b) ASTM tests. Y-axis individual value is (a) 10^{-14} m^2 and (b) 10^{-12} m^2. X-axis is the test number. UCL is the upper control limit based on the data. LCL is the lower control limit based on the data. $\bar{X}$ is the average of the data set and 1SL is the standard deviation from the mean of the data set.

In addition to the determining the feasibility and accuracy of each test, different modifications and techniques were recorded. These findings were summarized into standard operating procedures (SOPs) and given to Toray Advanced Composites for future use by their engineers and technicians (Appendix A and B). Some of these findings included:

Cirrus

- Calibrate the COHO mass flow meter by attaching it to a bagging apparatus with zero flow.
- The most common source of leaks exists at the crossovers of sealant tape strips. Using a straight edge or blunt tipped object was the easiest way to get rid of leaks.
- Set up the vacuum bag taught on the diagonal corners first for easiest application.

ASTM

- The recommended 10 grams of epoxy is sufficient for 3-4 samples.
- Sealant (chromate) tape should be used between the mounted sample and the apparatus to ensure an air-tight seal.
- A common source of leaks was improper filling of the epoxy between the acrylic blocks. This problem was resolved by applying an even amount of pressure when mounting the prepreg onto the acrylic blocks.

In addition to this data, other qualitative methods were used to determine which test is more feasible in the long-term. Based on the approximate decreased amount of resources required to run the ASTM test on one sample compared to the Cirrus test: 2 hours less, and 45 in^2 less. Additionally, four samples can be prepared simultaneously using the ASTM method whereas only one sample can be run at a time using the Cirrus method. Thus, the ASTM test was deemed more efficient.

CONCLUSIONS

1. Further modifications to the Cirrus and ASTM D8132 permeability testing methods are possible to make them more efficient and accurate.
2. The ASTM test was determined to be the preferred method for future testing due to lower amount of resources required.
3. Future work is required to determine the effect of out-time on the permeability of the different composite prepreg systems.

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[12] "Standard Test Method for Determination of Prepreg Impregnation by Permeability Measurement," pp. 1-8, 1 August 2017.

[13] T. Tedlund, "Gas Evacuation in Out of Autoclave Pre-impregnated Laminate," Grand Forks, ND, 2019.

APPENDIX

A. TORAY STANDARD DOCUMENT CONTROL PERMEABILITY CIRRUS METHOD

Testing of Permeability – Cirrus Method

Document Number

Department	Name	Signature	Date
Originator	Amy Lautenbach		
Expert Services	Amy Lautenbach		
Prod Engineering	N/A		
Procurement	N/A		
Manufacturing	N/A		
Quality	Wilson Vu		
EH&S	N/A		
Trainer	N/S		

Revision Record

Effective/Revision Date	Revision	Change	Existing Inventory
	NC	Initial Release	
		-	

1. Objective

1.1. This test standard describes the procedures for permeability testing as defined by Cirrus Aircraft

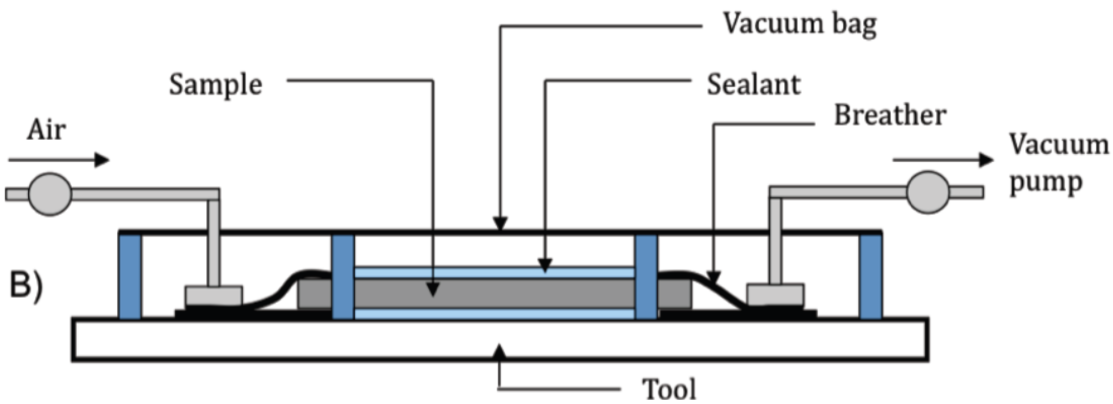
1.2. References:

1.2.1. Bagging layups

2. Specimens, Equipment and Apparatus

2.1. Specimens: 4 plies dimension 3.38” x 3.85” are laid up on top of each other and debulked for 1 hr. for EACH specimen (Example: For 3 tests you will need 12 plies measuring 3.38” x 3.85”)

2.2. Equipment: Sealant tape, breather cloth, vacuum ports, bagging material, vacuum (-25inHg) and mass flow meter



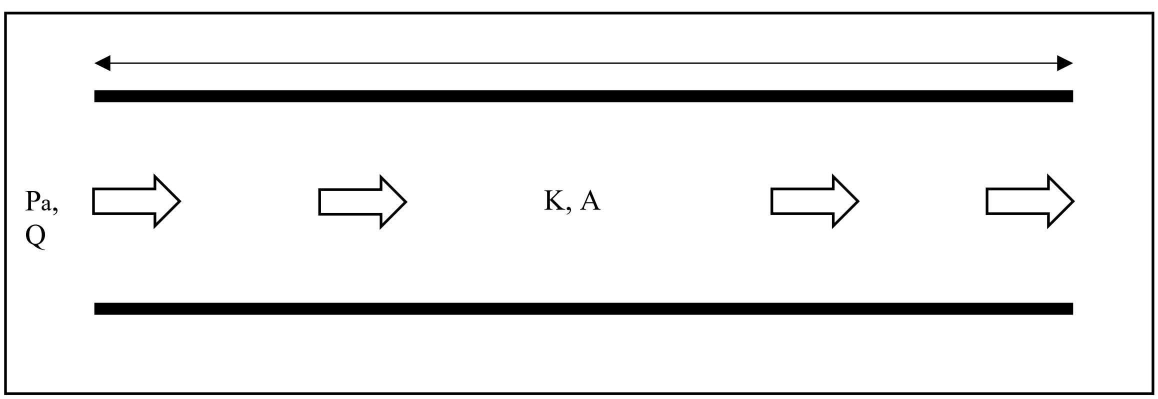


Figure 1: Airflow schematic of the CDC test, where Q is measured with a mass flow meter, P_0=101325 Pa, and P_L=14970 Pa.

3. Process

3.1. Cut 4 plies measuring 3.38” x 3.35” and lay up on top of each other oriented in the 0° direction.

3.2. Debulk 4-ply layup for 1 hour at -25 inHg (Debulk layup LU-1.5).

3.3. Set up chromate tape and breather on clean tool as illustrated in Figure 2. Outer dimensions are ~13”x4.” Also include 4” strips that run along the short length of the set up approximately 2” apart in the center. Leave paper on chromate tape. Place breather on either side of the chromate tape. Breather should not touch chromate tape.

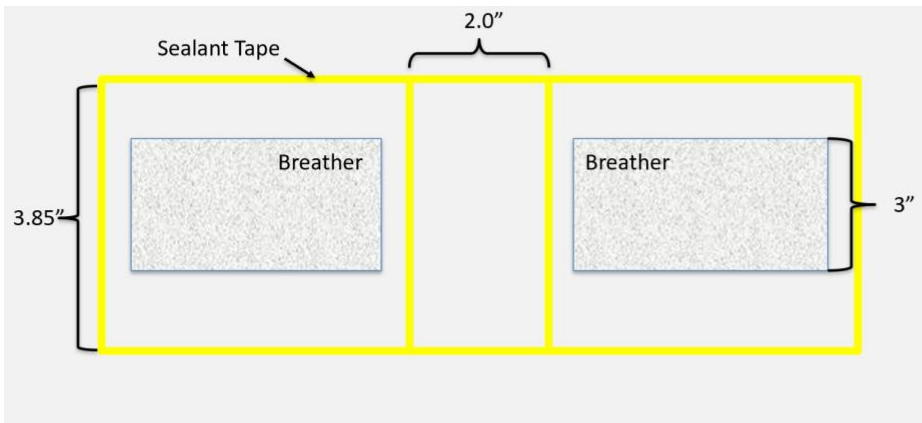


Figure 2: CDC setup for the sealant tape structure and breather material.

3.4. Peel off paper for chromate tape in the center. Place laminate on the chromate tape. There should be a slight overlap between the breather and composite. The shorter end of the laminate should not touch the border chromate tape (Figure 3).

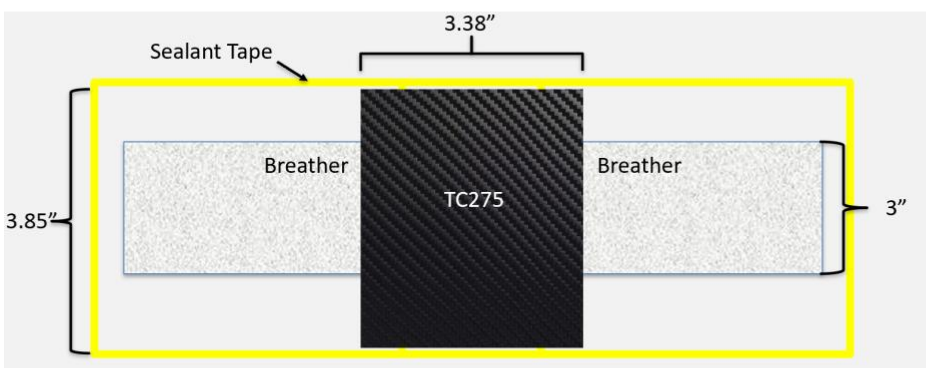


Figure 3: Debulked laminate placed on top of breather material and the two sealant (chromate) tape strips.

3.5. Place a second layer of chromate tape and breather on top of the laminate directly above the first layer of chromate tape and breather (Figure 4).

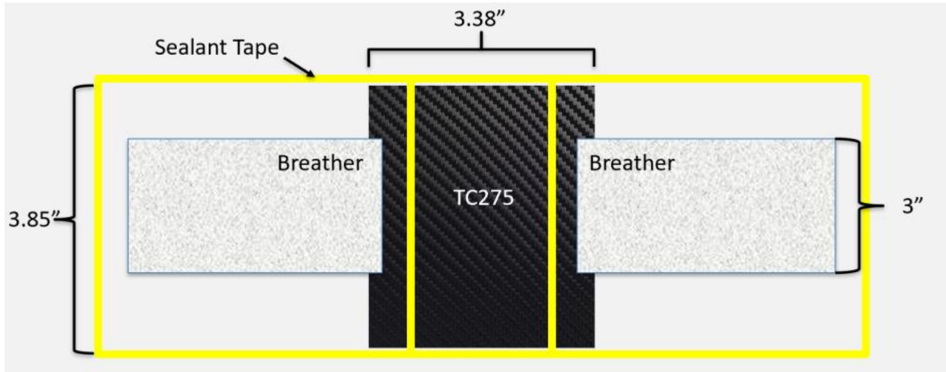


Figure 4. Two additional breather material pieces and sealant tape strips are placed on top of debulked laminate.

3.6. Place the bottom attachment of the vacuum Quick Connect port on the breather (outside the overlapping portion). See Figure 5.

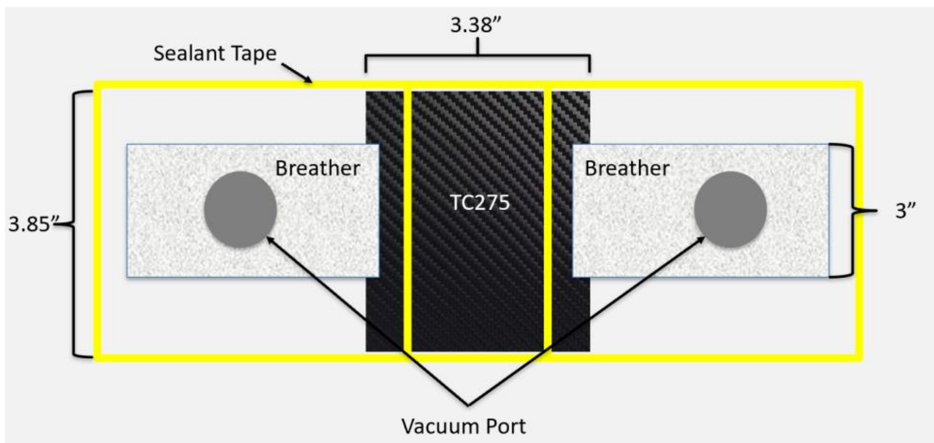


Figure 5. Vacuum ports placed solely on top of breather material.

3.7. Peel off remaining paper from chromate tape and lay bagging material over entire apparatus as taught as possible.

3.7.1. Recommendation: Place opposing diagonals first. Use a straight edge to completely seal bagging material to chromate tape.

3.8. Cut circular holes with a blade on vacuum port base as to attach the tops of the vacuum ports. Attach top of vacuum Quick Connect ports.

Figure 6. Complete apparatus before vacuum is applied.

3.9. Check for leaks in apparatus by pulling vacuum for a brief time (~30 seconds) and turning off the vacuum.
   3.9.1. Listen for leaks while vacuum is off. Bagging material will decompress in the presence of leaks.
   3.9.2. Replace one vacuum with a gauge. If the vacuum level is decreasing, there is a leak.
   3.9.3. Use a straight edge to fix leaks. Leaks are often found where different pieces of chromate tape intersect.

3.10. Vacuum is pulled to -25 in Hg and held for 1 hour.

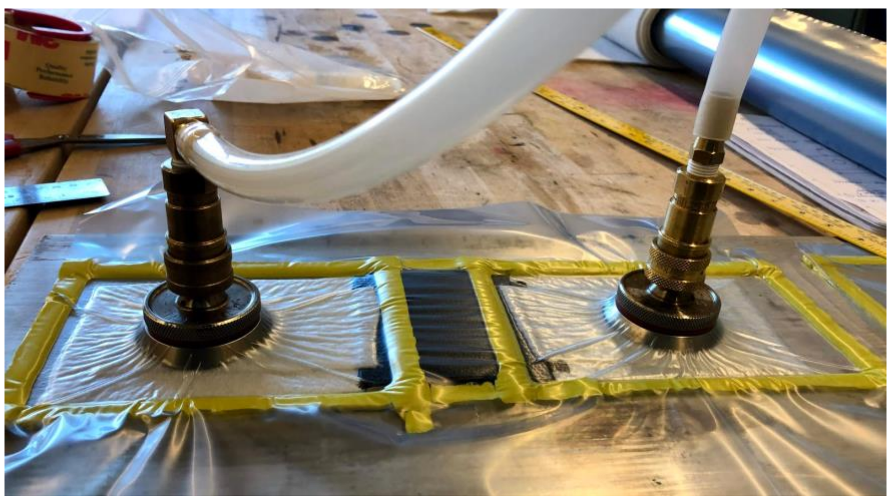
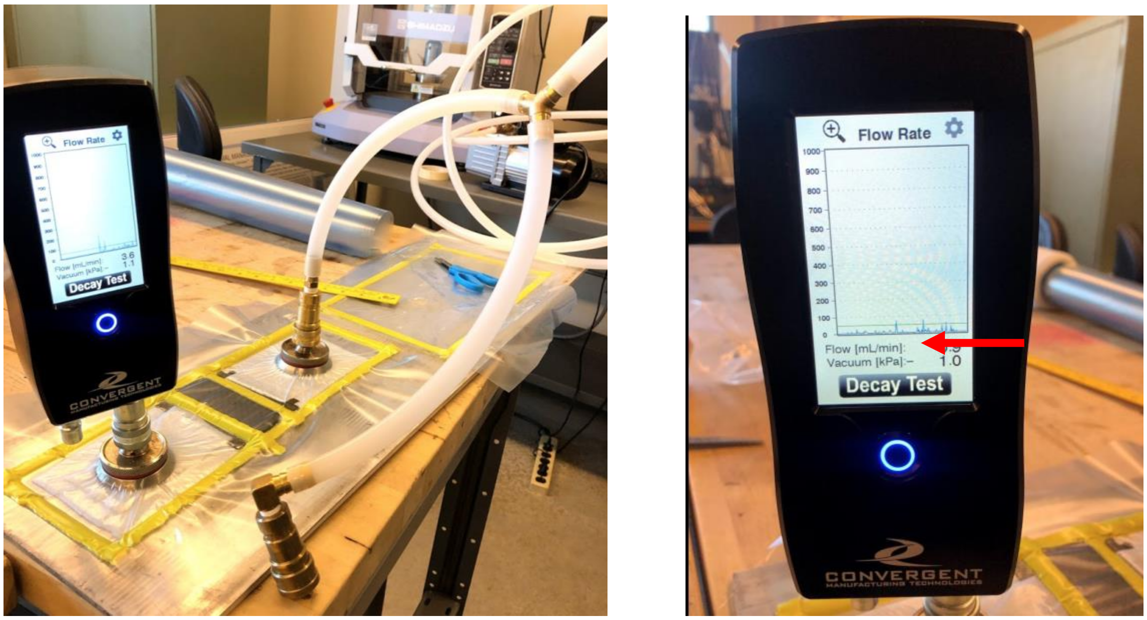


Figure 7. Vacuum applied to the apparatus. Note the compression of the bagging material.

3.11. After 1 hour: Detach one vacuum Quick Connect and attach the mass flow meter. One side should be attached to vacuum and the other to mass flow meter. Once volumetric flow is stabilized, collect the volumetric flow rate every minute for 10 minutes.



(a) (b)

Figure 9. (a) Testing apparatus. (b) Record mass flow readout indicated by red arrow.

4. Analysis

4.1. Equation: $K = \frac{2Q\mu L}{A}\left(\frac{\text{Po}}{\text{Po}^2 - \text{PL}^2}\right)$

K is the permeability (m^2)
Q is the average calculated volumetric flow rate (m^3/s)
µ is the dynamic viscosity of air (Pa*s)
L is the length of the laminate (m)
A is the cross-sectional area of the laminate (m^2)
Po is the absolute pressure of the atmosphere (Pa)
PL is the absolute pressure of the vacuum (Pa)

APPENDIX

B. TORAY STANDARD DOCUMENT CONTROL PERMEABILITY ASTM D8132

Testing of Permeability – ASTM D8132

Document Number

Department	Name	Signature	Date
Originator	Amy Lautenbach		
Expert Services	Amy Lautenbach		
Prod Engineering	N/A		
Procurement	N/A		
Manufacturing	N/A		
Quality	Wilson Vu		
EH&S	N/A		
Trainer	N/S		

Revision Record

Effective/Revision Date	Revision	Change	Existing Inventory
	NC	Initial Release	
		-	

5. Objective

5.1. This test standard describes the procedures for permeability testing per ASTM D8132. This test method determines the in-plane permeability of composite prepreg (pre-impregnated) materials as a measure of the level of impregnation.

5.2. References:

5.2.1. ASTM D8132 – Standard Test Method for Determination of Prepreg Impregnation by Permeability Measurement

6. Specimens, Equipment and Apparatus

6.1. Test Apparatus: Test apparatus is built according to drawing in Figure 1. A completed apparatus mounted to a baseplate is shown in Figure 2.

6.1.1. Equipment: Vacuum pump, vacuum hose, digital stopwatch, caliper, barometer, acrylic blocks, adhesive, and chromate tape.

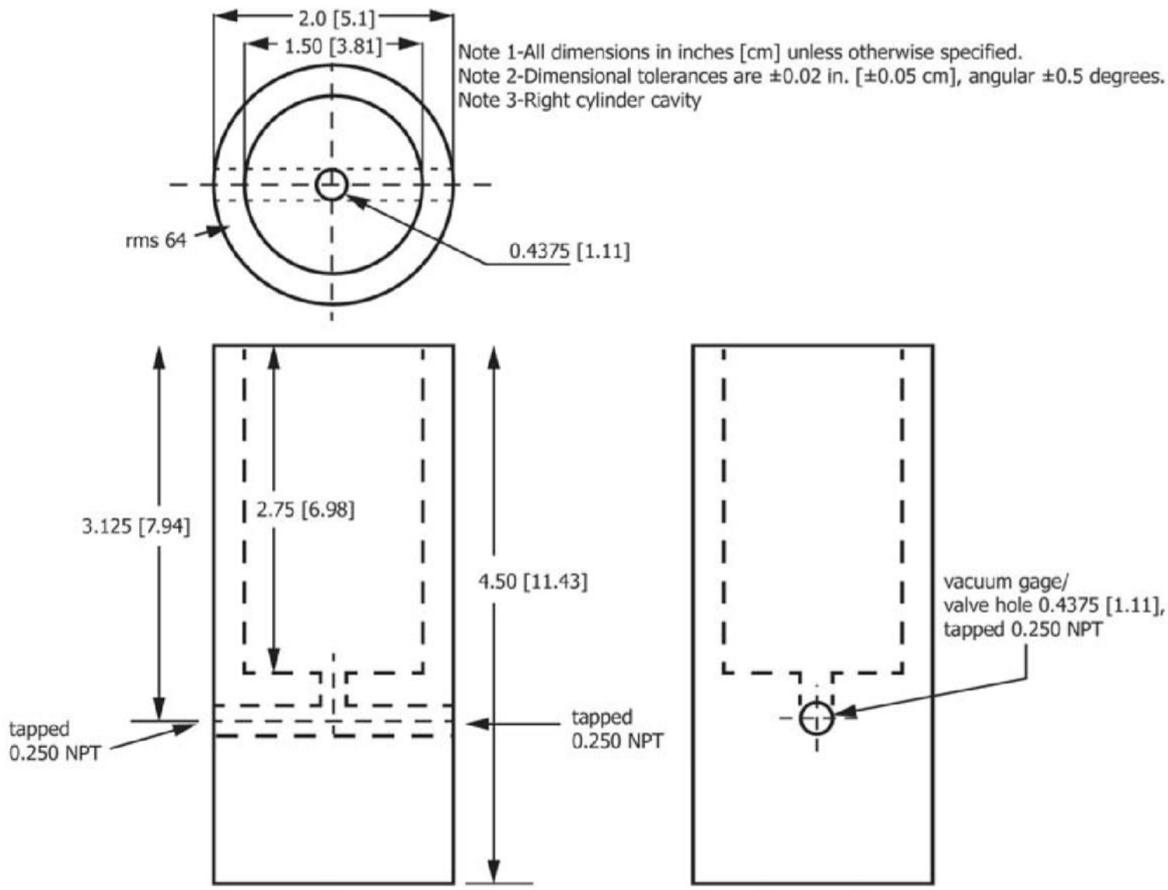


Figure 1. Apparatus design per ASTM D8132.

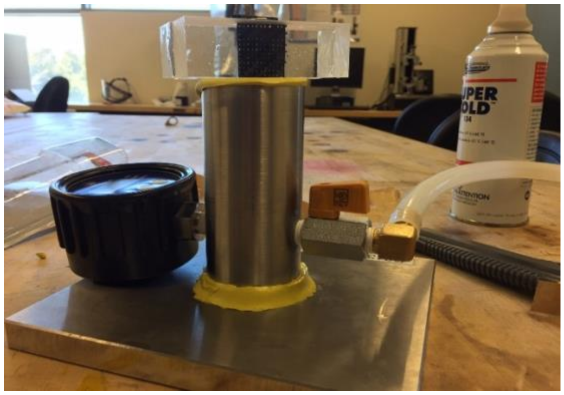


Figure 2. Complete apparatus from ASTM D8132 (Shown mounted to a base plate for stability)

6.2. Specimen:

6.2.1. Prepreg test specimens shall be 3.0" x 1.0" with the 3.0" in the 0° direction. When cutting, avoid pressing and compacting the prepreg surface. This is best done using Kevlar shears. Record width and thickness for each specimen to four significant figures in units of centimeters.

6.2.2. To produce the test specimen, the prepreg sample must be mounted between acrylic blocks to form a flat planar surface that can attach to the apparatus. Up to 3 samples may be prepared simultaneously.

6.2.2.1. Obtain two clean acrylic blocks. Each block should be 1" x 1" x 3" cut from stock acrylic bars as described in subsection 7.7 of ASTM D8132. Label with prepreg sample identification.

6.2.2.2. Mix approximately 10 g of epoxy adhesive in a clean plastic disposable container according to manufacturer directions as described in subsection 7.8 of ASTM D8132. (10 g is sufficient for 3 samples).

6.2.2.3. Spread a thin and consistent layer on one face of each block.

6.2.2.4. Carefully remove release liner from prepreg sample (hold at ends being careful not to compress prepreg) and place in the center on top of one adhesive-coated block. The prepreg and acrylic block should be perpendicular to each other. The prepreg should extend over each the edge of the block one inch.

6.2.2.5. Place the other adhesive coated block on top and apply contact pressure to form a good bond and an airtight seal. Wipe off any excess resin. Allow to cure fully over cup or similar device with ~ 2" diameter (Figure 3).

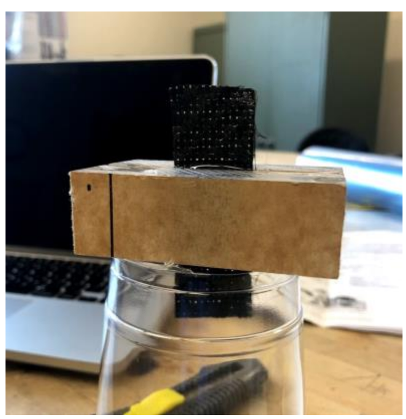


Figure 3. Mounted sample left to dry for an hour suspeneded on a plastic cup.

6.2.2.6. Before attaching to the apparatus, cut both ends of the prepreg as straight as possible without damaging the specimen. Measure the length of the prepreg to four significant figures.

7. Process

7.1. Calibrate the test vacuum chamber apparatus to ensure that it can maintain adequate vacuum level before testing.

7.1.1. Place sealant tape around the rim of the open, hollow cylinder and place a solid block on the sealant tape.

7.1.2. Apply full vacuum for one minute and then remove vacuum. Record the level of vacuum deduction over 30 minutes. The deduction must be less than 0.25 in Hg.

7.2. Record the temperature and humidity of the facility during testing as well as the actual barometric pressure.

7.3. Place new sealant tape on the apparatus top surface taking care to minimize the overlap beyond the interior edge of the hollow cylinder.

Figure 4. Sealant tape placed on the rim of the cylinder to allow for mounted sample to attach to the apparatus.

7.4. Place and center the cured specimen assembly onto the sealant tape located on top of the open cylinder.

7.5. Turn on and zero vacuum gauge.

7.6. With the valve in the open position, turn on the vacuum pump and start timer. After 1 minute, record the stabilized pressure.

7.6.1. Failure of pressure to stabilize indicates leaks. Push acrylic block down to ensure an airtight seal between the samples, chromate tape, and apparatus.

7.7. Close the vacuum valve. Record the pressure as a function of time in increments of 1 minute for 10 minutes.

7.8. After testing is complete, open the valve to equilibrate pressure, remove the sample, and remove sealant tape.

8. Analysis

8.1. Equation from ASTM D8132

$$\ln\left[\frac{(P_{atm}+P)(P_{atm}-P_i)}{(P_{atm}-P)(P_{atm}+P_i)}\right]=\frac{KWHP_{atm}}{LV\mu}t$$

P_{atm} is atmospheric pressure (Pa)
P_i is the initial pressure (absolute) in the vacuum chamber apparatus (Pa)
P is the pressure (absolute) in the vacuum chamber apparatus as a function of time (Pa)
K is the permeability (cm^2)
W is the width of the prepreg sample (cm)
H is the thickness of the prepreg sample (cm)
L is the length of the prepreg sample (cm)
V is the total volume of the vacuum chamber apparatus (cm^3)
μ is the viscosity of air (Pa-s)
t is the time

8.2. Graph permeability gradient value as a function of time (P is measured value).

$$\boxed{K}t=\underbrace{\ln\left[\frac{(P_{atm}+P)(P_{atm}-P_i)}{(P_{atm}-P)(P_{atm}+P_i)}\right]}_{\text{Permeability Gradient Value}}\underbrace{\frac{LV\mu}{WHP_{atm}}}_{\text{Constant}}$$

8.3. To calculate permeability K, multiply slope of permeability gradient value vs. time by constants.