# Moisture Characterization of Thermoplastics and Thermosets for use as Matrices in Fiber Reinforced Composites

**A Senior Project** 

Presented to the Faculty of the Materials Engineering Department California Polytechnic State University, San Luis Obispo

> In Partial Fulfillment of the Requirements for the Degree Bachelor of Science, Materials Engineering

> > by

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### Abstract

Epoxy matrix composites are widely used in aerospace industry for lighter aircraft while thermoplastic-matrix composites have traditionally been underutilized despite their excellent fracture resistance, impact strength, and the ability to be recycled. Toray Advanced Composites (Morgan Hill, CA) wants to investigate the structural integrity of thermoplastic matrices in common aircraft operating environments. An experiment was conducted to observe the effect of moisture absorption on thermoplastic composites' mechanical strength. PEEK, PPS, and PEI matrices were compared to an epoxy matrix in this experiment. Samples were submerged in 160°F distilled water to accelerate moisture absorption. For two months, the changes in mass and dimension were recorded at frequent intervals to compare the absorption characteristics of each matrix. Short-beam shear tests were conducted weekly to examine moisture's effect on interlaminar shear strength. Results indicated that epoxy had significantly higher saturation point and dimensional strain compare to the thermoplastics. At saturation, epoxy also had a greater decrease in interlaminar shear strength (34.0%) than the thermoplastics (PEEK: 9.11%, PPS: 16.24%, PEI: 10.18%). The coefficient of moisture expansion (CME) for each material was the slope of a linear trendline through a percent moisture content versus percent strain plot. Epoxy had the highest CME of 1.075, followed by PPS: 1.012, PEI: 0.532, and PEEK: 0.192. Although not directly proportional, higher CME seemed to correlate with higher strength loss.

**Keywords:** Materials Engineering, thermoplastic, thermoset, moisture, composites, short-beam shear, carbon fiber, PEEK, PPS, PEI, epoxy, coefficient of moisture expansion, hygric expansion, interlaminar shear, water absorption

### Acknowledgements

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### Introduction

#### **Problem Statement**

Composites that use a thermosetting polymer matrix are commonly used in high performance applications such as the aerospace industry. Thermoplastic matrices, on the other hand, are gaining momentum in the composites field due to their higher toughness. However, the effect of moisture absorption is not as well known in thermoplastics as it is in thermosets. Toray Advanced Composites (Morgan Hill, CA) is sponsoring this project to investigate moisture absorption in thermoplastics. Toray produces composite materials for companies in the aerospace, automotive, and space industries. They would like to provide accurate information to customers about their high toughness thermoplastic composites. However, the adverse effects of moisture absorption in thermoplastics needs to be investigated beforehand. This project compares the response to moisture of three thermoplastic matrices to a representative thermoset (epoxy). The three thermoplastics chosen were: polyether ether ketone (PEEK), polyetherimide (PEI), and polyphenylene sulfide (PPS). The results from this project will help Toray Advanced Composites make better informed material selection decisions for their clients in industries that encounter moisture such as aerospace.

#### Background

Since the introduction of carbon fiber reinforced composites, the world of ultralight, ultra-strong, and high stiffness components has been completely revolutionized. Carbon fiber has properties that make it one of the best materials for these applications. Along the direction of the fiber, carbon can have one of the highest specific stiffness and specific strengths of any material. However, carbon fiber on its own is virtually useless for most real-world applications. The fibers on their own, which are ten times thinner than a human hair, are easily buckled and therefore incapable of supporting any load in compression. Carbon gets all the credit for its properties, but the other side of the story is in the matrix.

When a composite is made, the fibers (most commonly carbon) are typically held together by a polymer matrix. This polymer can be thought of as a glue in that it adheres the fibers together and forms a cohesive material. However, the matrix is never as strong or stiff as the fibers. What the matrix does is transfer load to the fibers, which are much more capable of bearing the load. This requires that the matrix have the right properties as well as the right

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processing to adhere well and be distributed between all the fibers. One factor that can adversely affect composite parts is the humidity of the environment which can cause the matrix to swell.<sup>1</sup> Fortunately, the most common matrix materials are thermoset epoxies which have well documented responses to humidity. However, the composite industry is interested in moving towards new matrix materials: thermoplastics. Thermoplastics are structurally different than thermosets and have many structural benefits such as a higher toughness. What is not well known about thermoplastics is their response to moisture. The aim of this senior project is to investigate the effects of moisture absorption on thermoplastic matrices.

#### Importance of Composites in Industry

Fiber-reinforced composites offer a combination of strength and modulus that are either as good as or better than those of metallic materials. Their low density, high fatigue strength, and corrosion resistance make them superior materials in aircraft, space, automotive and marine applications. From boron fiber-reinforced epoxy skin of F-14's horizontal stabilizer in 1969, carbon-fiber reinforced epoxy wings, aileron and forward fuselage of AV-8B in 1982, to Airbus A380's 25% by weight composites content in 2006 (Figure 1), durability and structural integrity of composite materials have been well established.





Today, there are three main types of composites being used in aircrafts: carbon fiber-, glass-, and aramid- reinforced epoxy. As the primary choice of matrix material, epoxy is favored by the aerospace industry due to its low manufacturing cost, chemical resistance and thermal resistivity. However, its low toughness resulted in their low impact strength, and periodic inspection of composite components often shows damage caused by ground handling incidents,

foreign object impacts, and lightning strikes. On the other hand, thermoplastic-based composite materials were not considered major structural materials until recently because thermoset composites' properties have been well explored by the industry for many years. Many companies have already invested in capital equipment for thermoset processing. However, the desired properties such as low porosity content, weldability, high impact strength, and fracture toughness of thermoplastic composite materials have the potential to revolutionize the aerospace industry. <sup>[2]</sup> Before that can happen, further research is required on the interaction between the behavior of thermoplastic composites and the thermal and hygric environments that aircrafts commonly experience.

#### Thermoset Matrices

Thermosets are the most common polymer currently used in composites. A thermosetting polymer is a plastic that forms by a chemical reaction which causes cross-links between molecular chains to form, making it rigid. One common example of thermosets are epoxies. Epoxy was prevalence in the aerospace industry for their low processing cost and manufacturability. It possesses fair mechanical strength, high chemical, and thermal resistance thanks to the hydrogen bonding dipole moment generated by the hydroxyl group in its polymer structure (Figure 2).<sup>[3]</sup> Epoxies are named for their epoxide functional group, which allows them to form cross-links with other polymer chains. At elevated temperatures, or with the addition of hardening compounds, epoxy will harden from a liquid resin into a rigid plastic. This is used in the processing of composites and is what allows composites to be molded into various shapes. Liquid epoxy resin is added to fibers or a fabric, allowed to penetrate the fibers, then a vacuum is typically applied, and it is allowed to cure. In high performance composites, pre-impregnated (prepreg) plies are imbued with partially-cured epoxy resin and stored in a freezer. The kinetics of the chemical reaction prevent the epoxy from hardening until it is brought to an elevated temperature. When ready, these sticky prepreg lamina are adhered and put under vacuum. Curing is commonly done in an autoclave, which is essentially a high-pressure oven. The pressure, in addition to an applied vacuum, allows excess resin and any voids or air bubbles present to escape, resulting in an extremely light and strong part.

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Figure 2. The chemical structure of epoxy showing crosslinks and hydroxyl groups, which contribute to their desirable properties.

#### Thermoplastic Matrices

In contrast to thermosets, thermoplastics are not made from cross-linking a liquid precursor into a solid plastic. Instead, thermoplastics are solid and must be melted to be used as a composite matrix. Thermoplastic prepreg is a solid, therefore it is not sticky like a thermoset prepreg. This affects the processing, because each ply must be held in place by melting small tack regions using a heated element such as a hot rod. Then, instead of using a vacuum or pressurized gas, a mechanical press is used to apply heat and pressure to the composite. While in an autoclave, thermosets are cured through the acceleration of a chemical reaction. In the case of thermoplastics, no chemical reaction is taking place but rather the plastic is physically melting in order to adhere layers together. <sup>[3]</sup>

One benefit to thermoplastics is that they are potentially recyclable, unlike thermoset matrix composites. This is potentially important in the composites industry because currently most composites are not recyclable. Another benefit is that thermoplastic composites can be repaired. If a crack or dent is found in a thermoplastic composite part, it can essentially be heated up and reshaped an infinite number of times. A third benefit to thermoplastic composites is that they have a much higher toughness than thermoset composites. Thermosets are brittle, meaning they will not permanently deform without fracturing. On the other hand, thermoplastics are often extremely tough, meaning they can absorb much more energy than thermosets. This is especially important if a composite ever encounters an impact force. Impacts can cause brittle cracks to form in composites, because the energy the material can absorb without cracking is low. If a composite was made with a thermoplastic matrix, it could withstand an impact without significantly losing strength, because the ductility and toughness of the polymer absorbs some of

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the energy from impact. PEEK, PPS, and PEI, are all considered high performance thermoplastics for they exceptional mechanical strength, thermal, and chemical stability, which are the result of their aromatic polymeric backbone (Figure 3).



Figure 3. The aromatic rings compose of the backbone for a.) PEEK, b.) PPS, and c.) PEI, providing them with high stiffness and resistivity to thermal and chemical degradation. <sup>[3]</sup>

#### Moisture Absorption

Water molecules are absorbed by most polymers due to hydrogen bonding.<sup>[4]</sup> In some plastics this causes the glass transition temperature to decrease, making the plastic softer and weaker. The other resulting effect of moisture is that it causes polymers to expand. This hygric swelling can be quantified with the coefficient of moisture expansion (CME).

In the case of composites, the CME is sometimes categorized into longitudinal and transverse values, denoted as  $\beta_1$  and  $\beta_2$  respectively. The CME is found by taking the slope of a linearized data for hygric strain versus moisture content (Figure 4).



Figure 4. Plot of hygric strain versus moisture concentration in composite test samples. The slope of the linear trendline is taken as the coefficient of moisture expansion <sup>[1]</sup>

The difference between the CME transverse to the fibers versus along the direction of the fibers is caused by the anisotropy of the composite interacting with the expansion of the matrix; the orientation of the fibers restricts the expansion in different directions. These values are important to know when processing and designing a composite part. If a part changes dimensions during the manufacturing process due to moisture, it could have an adverse effect on the end use which must be accounted for. Because thermoplastics and thermosets are fundamentally different in their polymer structure, it is likely that their response to moisture will be different.

#### Short-Beam Shear

The goal of the short-beam shear test is to evaluate the interlaminar shear strength of a composite laminate. The test used in this experiment were based around ASTM D2344. In this test, a relatively thick composite sample is placed between three rollers with a span ratio of 4:1 with the sample thickness (Figure 5).



Figure 5. Interlaminar shear schematic illustrating the short-beam shear test. A short span width causes beam shear which leads to interlaminar delamination as the failure mode. <sup>[5]</sup>

Based on the mechanics of this loading arrangement the sample is put in shear and will fail with the delamination of each ply. This is predominantly a matrix-dominated property of the composite, because the matrix properties will determine the adhesion between plies. The hypothesis of this experiment is that if moisture absorption has an impact on matrix strength, then the interlaminar shear strength will be affected.

### **Experimental Methods**

#### Safety

Standard lab safety procedures were followed. Since carbon fiber filaments in composite panels were micron-scale in size, in addition to regular laboratory attire (close-toed shoes, long pants, and goggles) nitrile gloves were worn when handling the samples to prevent skin irritation and injuries. While in the lab, the experimenters were always accompanied by a partner in order to prevent and react to any unforeseen danger.

#### Sample Preparation

Four carbon fiber composite panels were fabricated with the help of Toray Advanced Composites. The fiber constituent was AS-4 carbon fiber, which is a PAN-based carbon fiber commonly used by the aerospace industry, and the matrix constituent for each sample panel was TC250E-6 epoxy, PEEK, PPS and PEI. The epoxy panel was processed by vacuum-bag autoclave molding, and the laminate was comprised of 32 plies of pre-impregnated AS-4 fabric. On the other hand, because the thermoplastic panels curing process did not involve crosslinking, a hot press was used to bond the prepreg plies together. The lay-up for PEEK, PPS, and PEI laminates were as follows:  $[0^{\circ}/90^{\circ}]_{7s}$  for the PEEK sample,  $[0^{\circ}/90^{\circ}]_{8s}$  for the PPS sample, and  $[0^{\circ}/90^{\circ}]_{7s}$  for the PEI sample. The reason behind different number of plies for each sample was to achieve similar thickness. While difference in number of layers would affect the mechanical strength of each sample, the focus of this project was to examine the relative strength loss among all four samples instead of their absolute mechanical strength. Similarly, the difference between the fabric and cross-ply laminates would be negligible in this experiment because the matrix is the dominant factor that decides the strength loss due to moisture absorption. The laboratory request forms that contain detailed information about the fabrication of each sample panel are listed in Appendix A through D. Each sample panel was sectioned into 40 coupons which were roughly 1 inch in length before the moisture exposure took place. Within each sample of the same matrix, the coupons were assigned into 8 groups, A through H, of 5 for different submersion duration with group H being the control dry group. Within each alphabetic group, each coupon was assigned a number between 1 and 5 (i.e., A1-A5, B1-B5, and so on).

#### Moisture Exposure

ASTM D5229 was referenced to decide the moisture exposure method. <sup>[6]</sup> Due to the limited scope of this project and that the purpose of this project was to compare the apparent absorption properties and the relative strength loss among all samples, the moisture exposure was done by submerging the samples in a liquid bath. The moisture bath was filled with distilled water and held at 160°F (Figure 6). The moisture exposure continued until saturation was observed in every sample, which took a total of 62 days to accomplish. The moisture content and hygric strain of each sample was monitored by measuring the mass twice a week throughout the entire submersion period.



Figure 6. Moisture bath provided by Toray Advanced Composites. Samples were immersed in distilled water held at a temperature of 160°F.

#### Dimension Measurement

The thickness, length, and width of all 40 specimens of each sample were measured before the moisture submersion as reference for any hygric expansion. Dimension measurement was carried out using Mitutoyo micrometer series 293 twice a week. All five coupons of group G and the alphabetic group subjected to SBS test were pulled out of the moisture bath while only two coupons of each other alphabetic group were pulled out twice a week for the dimension measurement.

#### Short-Beam Shear Test

Short-beam shear testing was done in accordance to ASTM D2344<sup>[5]</sup> on the Instron 5584 using a fixture shown in (Figure 7). The SBS test was performed on all five coupons of one alphabetic group once per week. The test outputs load versus displacement, where the maximum load is taken to determine the interlaminar shear strength (ILSS).



Figure 7. Short-beam shear fixture mounted on the Instron 5584. The span width was set to a 4:1 ratio of span to thickness in accordance to ASTM D2344. <sup>[5]</sup>

## Results

Epoxy absorbed significantly more moisture than the thermoplastics did, reaching a saturation level of approximately 2.0wt% (Figure 8). Similarly, the change in thickness showed that epoxy experienced the most hygric strain, up to 2.5% at saturation (Figure 9). The data plotted on these figures is the average of approximately half of all samples for each matrix, and can be found in Table I and Table II for mass gain and hygric strain, respectively.



Figure 8. Plot of moisture content in weight percent versus immersion time in days. Epoxy, in blue, reaches saturation at approximately 2.0% moisture content. The thermosets, however, reach a much lower level of saturation and saturate earlier in the experiment. The standard deviation of each data point is shown using error bars, which show fairly low variance.

# of Days	Ероху	PEEK	PPS	PEI
3	0.32%	0.30%	0.08%	0.23%
7	0.59%	0.29%	0.09%	0.32%
10	0.71%	0.31%	0.16%	0.30%
16	0.89%	0.27%	0.10%	0.41%
29	1.35%	0.36%	0.22%	0.52%
32	1.55%	0.38%	0.29%	0.43%
35	1.63%	0.35%	0.30%	0.41%
37	1.64%	0.36%	0.28%	0.39%
42	1.80%	0.34%	0.28%	0.40%
47	1.85%	0.35%	0.31%	0.43%
49	1.91%	0.34%	0.27%	0.40%
51	1.92%	0.35%	0.30%	0.46%
56	1.98%	0.35%	0.30%	0.43%
58	1.86%	0.30%	0.31%	0.42%
64	2.00%	0.30%	0.37%	0.44%

Table I: Percent Moisture Content by Mass and Number of Days for each Matrix



Figure 9. Plot of hygric strain in percent versus immersion time in days. Epoxy, in blue, experiences a significant amount of hygric strain compared to the thermosets. PEEK, especially, shows virtually no expansion due to moisture. The error bars show the standard deviation of each data point and are wider than the mass measurements due to the accuracy of the micrometer used.

# of Days	Ероху	PEEK	PPS	PEI
3	0.33%	0.10%	0.19%	0.16%
7	0.56%	0.07%	0.19%	0.14%
10	0.74%	0.08%	0.26%	0.21%
16	0.85%	0.07%	0.26%	0.20%
29	1.32%	0.00%	0.19%	0.21%
32	1.52%	0.01%	0.21%	0.15%
35	1.61%	0.05%	0.21%	0.16%
37	1.75%	0.10%	0.35%	0.25%
42	1.93%	0.11%	0.29%	0.26%
47	2.05%	0.03%	0.32%	0.24%
49	2.11%	0.06%	0.28%	0.22%
51	2.20%	0.08%	0.36%	0.30%
56	2.25%	0.08%	0.23%	0.23%
58	2.38%	0.06%	0.32%	0.21%
64	2.48%	0.08%	0.35%	0.29%

 Table II: Percent Hygric Strain and Number of Exposure Days for each Matrix

Using a linear trendline, fit to the data of moisture content versus hygric strain, we can determine the coefficient of moisture expansion (CME) which is taken as the slope of the trendline (Figure 10). Since the thermoplastics absorbed much less moisture than epoxy, it is more difficult to confidently put a value on their CME (Figure 11). Epoxy had a CME of 1.075 (p<0.001), PPS

had a CME of 1.012 (p=0.005), PEI had a CME of 0.532 (p=0.0013), PEEK had a CME of 0.192 but was not statistically significant (p=0.306).



Figure 10. Percent hygric strain versus moisture content. Slope of the trendline is taken as the coefficient of moisture expansion (CME). Epoxy has a more clearly defined slope due to reaching a higher saturation level of moisture content.



Figure 11. Close up of percent strain versus moisture content, showing CME for thermoplastics. Due to more considerable scatter than epoxy, the CME for the thermoplastics has less statistical significance. However, a comparative analysis is still possible.

The CME was also attempted to be determined in the longitudinal direction of the fibers but was inconsistent due to the small changes in dimension observed. The change in length was too small to be accurately measured, but shows PEEK and PPS experiencing negative strain (Figure 12). Similarly, the change in width was too small for the accuracy of the micrometers (+/- 0.001mm) used and was therefore not used to determine the CME (Figure 13).



Figure 12. Change in length of samples versus immersion time in days. Due to extremely small changes in length and limited accuracy of the micrometers used, no conclusions could be made from this data. The strength of the fibers in the longitudinal direction clearly restricts the expansion of the matrix.



Figure 13. Change in width versus immersion time in days. Similar to the changes in length, significant variance prevents any further analysis from being done. However, it does appear that PEEK and PPS may experience a negative expansion in the longitudinal direction of the fibers.

Based on short-beam shear testing, epoxy experienced the greatest decrease in interlaminar shear strength (ILSS) over the course of the experiment (Figure 14). The thermoplastics, on the other hand, did not lose as much ILSS due to the lower saturation levels they reached. Due to considerable variation of strength throughout the testing phase, the average of the ILSS was taken after each material reached saturation for comparison to the CME (Table III).



Figure 14. Interlaminar shear strength versus exposure time. There is a significant amount of variation both in the standard deviation of each data point, shown with error bars, and the change with time. Due to this, the average was taken from all data points following saturation of each matrix.

Matrix:	Ероху	PEEK	PPS	PEI
Loss in ILSS:	34.0%	9.11%	16.24%	10.18%
CME:	1.075	0.192	1.012	0.532

Table III: Loss in Interlaminar Shear Strength at Saturation and Coefficient of Moisture Expansion

### Discussion

The thermoplastics did not absorb nearly as much moisture as epoxy did. This is to be expected, due to epoxy having hydroxyl functional groups which experience hydrogen bonding with water molecules. The thermoplastics, on the other hand, do not have these polar groups, and therefore experience less moisture absorption. In addition to the increase in mass from moisture absorption, the hygric swelling the samples experienced represents the polymer expanding as water infiltrates it. As with moisture absorption, epoxy experienced the greatest degree of hygric expansion, while the thermoplastics did not swell nearly as much. PEEK, specifically, experienced a nearly negligible amount of hygric swelling. This is confirmed when we look at the data for the coefficient of moisture expansion (CME).

The CME data shows that epoxy and PPS are more sensitive to hygric swelling than PEI and PEEK. While the specific values for the CME were not statistically significant for PEEK, it is apparent that it has a lower CME than the other materials. The longitudinal CME, from width and length change, could not be accurately determined but appears to be negative for PPS and PEEK. This is possibly due to carbon fiber experiencing a negative hygric expansion, as has been observed in literature.<sup>[7]</sup> The CME can be useful in the manufacturing of composites in order to prevent warpage, but a more focused experiment is needed to determine more accurate values for this. What is relevant, however, is the relationship between the CME and the decrease in interlaminar shear strength (ILSS).

Interestingly, the materials that lost the least ILSS also had the lowest CME (PEEK and PEI). The converse is also true, epoxy and PPS lost the most ILSS and had greater CME than the other materials tested. This suggests that the CME may be a predictor of a matrix material's sensitivity to moisture related strength loss. Potentially, this could be applied to new materials as they are developed and prevent the need for strength testing if a relationship can be established.

The decrease in strength observed can be attributed to both the decrease in matrix strength with moisture, as well as the decrease in fiber interfacial strength in bonding to the matrix. It can be assumed that if compression or tensile testing were to be done that the decrease in strength would not be as dramatic as this experiment observed. This is because the short-beam shear test is matrix-dominated, whereas a compression or tensile test is mostly determined by the strength of the fiber. In this way, the decreases in strength that were observed can be taken as a worst-case scenario.

Ultimately, since the thermoplastic matrices outperformed the industry standard, epoxy, this experiment supports the feasibility of thermoplastics for use in humid environments. Due to limitations in this experiment's timeframe, it was not possible to examine whether cyclic exposure to moisture would have any unforeseen adverse effects, and because of this it is recommended that such an experiment be conducted. In addition, since thermoplastics are being transitioned to because of their superior toughness, it would be relevant to examine the effects of moisture on composite toughness in a future study.

## Conclusions

- 1. At saturation, the thermoplastics experienced a lower decrease in interlaminar shear strength than epoxy.
- 2. The matrices with higher coefficients of moisture expansion experienced a greater decrease in interlaminar shear strength.
- 3. Epoxy absorbed more moisture than the thermoplastics and reached a higher saturation level.

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## Appendix A

LAB	ORATORY REQUEST F	ORN	
Requestor Lautenbach, Amy P/O #	In-process	LR	<b>#</b> 28132
Index#	10 #	Di	ate 1/16/2019
Need Date	/0 #	Start D	ate 01/16/2019 -
Customer TCAC		Test En	d Date
Test Spec. TCAC Internal	Cert. #	Test Ti	me737075
Description BT250E-6/AS4C 3K PW, 195 gsm	40%RC TCAC# 50121862	Type Fabric	Cure Autoclave
Fiber/Fabric AS4C Area Wt. 19	Resin BT250E-6	Width 48	RC% 40
Sample Requirements	Box#	TAR#	Test Lead Time
	Material Lot # 010319-2T3		Project Code
	Resin Lot # TBD		Test Code Other
Pre-Test Preparation & Instruct	on:		
LAY-UP LU-1 with bleeder strings, Debulk even	/ 4-5 ply >15 minutes		
CURE	255		Hold
Vacuum <26 inHg, add 44 psi pressure minute. Cool <7°F/min to >150°F, rele	e, Heat 1 - 5°F/min to 260±10°f ase pressure and vacuum.	based on lagging	тс. нож 280-150
CSCAN			
MACHINE			
SBS per ASTM D2344, (tx2)" x (tx6)" -	length at least 1" $\Omega = 80$		
FV 1.0"x0.5" (n=3)	7		
TEST			
FV/VV/PPT (n=3)			
0° SBS per ASTM D 2344-84 @RTD, 3p	t, 4:1Span (n=5), 0.05"/min(SI	3>7.4) per lot	
Testing Data & Results:		~	LR# 28132
# Test °F Sample	Information Lot# Ray	v Norm. Ur	nit N-% Rprt #
			<u> </u>
2			
			•
Test Results			Form Rev: 3/1/10- BRM
		~	

## Appendix B

LABORATORY RE	QUEST FORM
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LR # 28133 Date 1/16/2019 Start Date 01/16/2019 Test End Date Test Time737075 60124444 Type Tape Cure Thermoplastic PEEK Width 6 RC% 34 TAR# Test Lead Time 1418-1TP1 2T Project Code b) Test Code Other thickness minimal over) ar, 20 min. and 10°C/min. cool)	P/O # With the second s	0 # Cert. # TCAC# <u>60124444</u> 5 Resin <u>PEEK</u>  Box# Material Lot # <u>041418-1TP1 2T</u> Resin Lot # <u>TBD</u> Dn:	L Start I Test E Test T Type Tape Width 6  TAR#	R # 28133 Date <u>1/16/2019</u> Date <u>01/16/2019</u> nd Date "ime737075 Cure Thermoplastic RC% <u>34</u> Test Lead Time Project Code Test Code Other
Date 1/16/2019         Start Date 01/16/2019 -         Test End Date	Index# With the second	D # Cert. # TCAC# <u>60124444</u> 5 Resin <u>PEEK</u> Box# Material Lot # <u>041418-1TP1 2T</u> Resin Lot # <u>TBD</u>	Test T Test T Test T Type Tape Width 6 TAR#	Date <u>1/16/2019</u> Date <u>01/16/2019</u> nd Date "ime737075 Cure Thermoplastic RC% <u>34</u> Test Lead Time Project Code Test Code Other
Start Date 01/16/2019	Need Date	TCAC# <u>60124444</u> 5 TCAC# <u>60124444</u> 5 Resin <u>PEEK</u> Box# Material Lot # <u>041418-1TP1 2T</u> Resin Lot # <u>TBD</u> DD:	Start I Test E Test T Type Tape Width <u>6</u>  TAR#	Date 01/16/2019 nd Date "ime737075 Cure Thermoplastic RC% 34 Test Lead Time Project Code Test Code Other
Test End Date         Test Time      737075         60124444       Type Tape       Cure Thermoplastic         PEEK       Width 6       RC% 34         TAR#       Test Lead Time         1118-1TP1 2T       Project Code         10       Test Code Other         11       Test Code Other         11       Test Code Other         11       Test Code Other         12       Test Code Other         13       Test Code Other         14       Test Code Other         14       Test Code Other         15       Test Code Other         16       Test Code Other         17       Test Code Other         18       Test Code Other         19       Test Code Other         10       Test Code Other         10 </th <th>Customer TCAC Test Spec. TCAC Internal Description AS4/PEEK Fiber/Fabric AS4 Sample Requirements al Poly Senior Project PPT 0.0054 Te-Test Preparation &amp; Instruction LAY-UP T1 - 28 ply (0/90)7s - 6"x6" (make panel CURE</th> <th>Cert. # TCAC# 60124444 TCAC# 60124444 Resin PEEK Box# Material Lot # 041418-1TP1 2T Resin Lot # 180 Dn:</th> <th>Test E Test T Type <u>Tape</u> Width <u>6</u>  TAR#</th> <th>nd Date Time737075  Cure Thermoplastic  RC% 34 Test Lead Time Project Code Test Code Other</th>	Customer TCAC Test Spec. TCAC Internal Description AS4/PEEK Fiber/Fabric AS4 Sample Requirements al Poly Senior Project PPT 0.0054 Te-Test Preparation & Instruction LAY-UP T1 - 28 ply (0/90)7s - 6"x6" (make panel CURE	Cert. # TCAC# 60124444 TCAC# 60124444 Resin PEEK Box# Material Lot # 041418-1TP1 2T Resin Lot # 180 Dn:	Test E Test T Type <u>Tape</u> Width <u>6</u>  TAR#	nd Date Time737075 Cure Thermoplastic RC% 34 Test Lead Time Project Code Test Code Other
Test Time      737075         60124444       Type Tape       Cure Thermoplastic         PEEK       Width 6       RC% 34        TAR#       Test Lead Time         11418-1TP1 2T       Project Code         10       Test Code Other         11       Test Code Other         11       Test Code Other         11       Test Code Other         11       Test Code Other         12       Other         13       Test Code Other         14       Topological Code         14       Topological Code         15       Test Code Other         16       Test Code Other         17       Test Code Other         18       Topological Code         19       Test Code Other         10       Test Code Other         10       Topological Code         10       Topological Code </th <th>Test Spec. TCAC Internal Description AS4/PEEK Fiber/Fabric AS4 Sample Requirements al Poly Senior Project PPT 0.0054  re-Test Preparation &amp; Instructio LAY-UP F1 - 28 ply (0/90)7s - 6"x6" (make pane CURE</th> <th> TCAC# 60124444 5 Resin PEEK Box# Material Lot # 041418-1TP1 2T Resin Lot # TBD</th> <th> Test T Type Tape Width 6  TAR#</th> <th>Time737075 Cure Thermoplastic RC% 34 Test Lead Time Project Code Test Code Other</th>	Test Spec. TCAC Internal Description AS4/PEEK Fiber/Fabric AS4 Sample Requirements al Poly Senior Project PPT 0.0054  re-Test Preparation & Instructio LAY-UP F1 - 28 ply (0/90)7s - 6"x6" (make pane CURE	TCAC# 60124444 5 Resin PEEK Box# Material Lot # 041418-1TP1 2T Resin Lot # TBD	Test T Type Tape Width 6  TAR#	Time737075 Cure Thermoplastic RC% 34 Test Lead Time Project Code Test Code Other
60124444       Type Tape       Cure Thermoplestic         PEEK       Width 6       RC% 34	Description AS4/PEEK Fiber/Fabric AS4 Area Wt. 146 Sample Requirements al Poly Senior Project PPT 0.0054 re-Test Preparation & Instruction LAY-UP F1 - 28 ply (0/90)7s - 6"x6" (make pane CURE	TCAC# 60124444 5 Resin PEEK Box# Material Lot # 041418-1TP1 2T Resin Lot # TBD Dn:	Type <u>Tape</u> Width <u>6</u> TAR#	Cure <u>Thermoplastic</u> RC% <u>34</u> Test Lead Time Project Code Test Code Other
PEEK       Width 6       RC% 34         TAR#       Test Lead Time         11118-1TP1 2T       Project Code         8D       Test Code Other         strickness minimal over)       Test Code In the code other         ar, 20 min. and 10°C/min. cool)       0.05″/min	Fiber/Fabric AS4 Area Wt. 140 Sample Requirements al Poly Senior Project PPT 0.0054 re-Test Preparation & Instruction LAY-UP F1 - 28 ply (0/90)7s - 6"x6" (make panel CURE	Box# Box# Material Lot # 041418-1TP1 2T Resin Lot # 180 Dn:	Width <u>6</u> TAR#	RC% 34 Test Lead Time Project Code Test Code Other
TAR#Test Lead Time Project Code 30 Test Code Other thickness minimal over) ar, 20 min. and 10°C/min. cool) 0.05″/min	Sample Requirements al Poly Senior Project PPT 0.0054 re-Test Preparation & Instruction LAY-UP T1 - 28 ply (0/90)7s - 6"x6" (make pane CURE	Box# Material Lot # 041418-1TP1 2T Resin Lot # TBD	TAR#	Test Lead Time Project Code Test Code Other
11418-1TP1 2T       Project Code         3D       Test Code Other         sthickness minimal over)	al Poly Senior Project PPT 0.0054 re-Test Preparation & Instruction LAY-UP F1 - 28 ply (0/90)7s - 6"x6" (make pane CURE	Material Lot # 011418-1TP1 2T Resin Lot # TBD		Project Code Test Code Other
thickness minimal over) ar, 20 min. and 10°C/min. cool)	re-Test Preparation & Instruction LAY-UP F1 - 28 ply (0/90)7s - 6"x6" (make pane CURE	Resin Lot # TBD		Test Code Other
thickness minimal over) ar, 20 min. and 10°C/min. cool) 0.05″/min	r <del>e-Test_Preparation &amp; Instruction LAY-UP</del> F1 - 28 ply (0/90)7s - 6"x6" (make pane CURE	n:		Test code Other
thickness minimal over) ar, 20 min. and 10°C/min. cool) 0.05″/min	re-Test Preparation & Instruction LAY-UP F1 - 28 ply (0/90)7s - 6"x6" (make pane CURE	on:		
thickness minimal over) ar, 20 min. and 10°C/min. cool) 0.05″/min	LAT-UP F1 - 28 ply (0/90)7s - 6"x6" (make pane CURE			
ar, 20 min. and 10°C/min. cool) 0.05″/min	CURE	al to fit frame and this area -		
ar, 20 min. and 10°C/min. cool) 0.05″/min	CURE	ei to rit frame and thickness m	inimai over)	
ar, 20 min. and 10°C/min. cool) 0.05″/min				
0.05″/min	Press process in picture frame (10°C/mi	in to 385 °C. 30 har 20 min a	and 10°C/min cos	(10
0.05″/min	Tess process in picture name (10 G/m			~,
0.05″/min	MACHINE			
0.05″/min	SBS per ASTM D2344			
0.05″/min				
0.05″/min	-v u.5 x2.0" (n=3)			
0.05″/min	TEST			
0.05″/min	EV/VV/PPT (n=3)			
, 0.05″/min	.,,			
	0° SBS per ASTM D 2344-84 @RTD, 3pt	t, 4:1 Span (n=5), 0.05″/min		
Checked by:	sting Data & Results:			Checked by:
t# Raw Norm. Unit N-% Rprt #	Test °F Sample I	Information Lot# Ra	w Norm. U	nit N-% Rprt #
ot# Raw Norm. U	sting Data & Results: Test °F Sample 1	Information Lot# Ra	w Norm. U	

## Appendix C

Requestor Lautenbach, Amy			
	Awaiting Mat.		
P/O # (21800224) PO-TEN-023 v1		L	R # 28134
Index# 95956	WO # WO-024116	D	ate 1/16/2019
Neéd Date		Start D	ate 1/16/2018 -
Customer TCAC		Test Er	d Date
Test Spec. TCAC Internal	Cert. #	Test Ti	ime736710
Description AS4/PPS, 115 gsm, 40% RC	TCAC# 61125094T3	Type Tape	Cure Thermoplastic
Fiber/Fabric AS4 Area Wt.	115 Resin PPS	Width 6.25	RC% 40
Sample Requirements	Box#	TAR#	Test Lead Time
al Poly Senior Project R#25904 RN 1675	Material Lot # 041618-1TP1 1T		Project Code
	Resin Lot # TBD		Test Code Other
re-Teet Branaration & Instrum	tion.		
LAY-UP		ADDT-	0.00399
		FIDE	
11 - 32 ply (0/90)8s - 6"x6" (make p	anei to fit frame and thickness mil	nimal ovér)	
CURE			
Press process in picture frame (10°C	/min to 310°C, 30 bar, 20 min. an	d 10°C/min. cool	)
MACHINE			
SBS per ASTM D2344			
FV 0.5"x2.0" (n=3)			
FV 0.5"x2.0" (n=3)			
FV 0.5″x2.0″ (n=3) TEST			
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3)			
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3)			
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD,	3pt, 4:1 span (n=5), 0.05"/min		
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD,	3pt, 4:1 span (n=5), 0.05"/min		
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD,	3pt, 4:1 span (n=5), 0.05″/min		
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD,	3pt, 4:1 span (n=5), 0.05″/min		
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD,	3pt, 4:1 span (n=5), 0.05″/min		
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD,	3pt, 4:1 span (n=5), 0.05"/min		10# 20124
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD, sting Data & Results:	3pt, 4:1 span (n=5), 0.05"/min		LR# 28134
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD, sting Data & Results: Test °F Sampl	3pt, 4:1 span (n=5), 0.05"/min	v Norm. U	LR# 28134 Checked by: nit N-% Rprt #
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD, sting Data & Results: Test °F Sampl	3pt, 4:1 span (n=5), 0.05"/min	v Norm. U	LR# 28134 Checked by: nit N-% Rprt #
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD, sting Data & Results: Test °F Sampl	3pt, 4:1 span (n=5), 0.05"/min	v Norm. U	LR# 28134 Checked by: nit N-% Rprt #
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD, sting Data & Results: Test °F Sampl	3pt, 4:1 span (n=5), 0.05"/min	v Norm. U	LR# 28134 Checked by: nit N-% Rprt #
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD, sting Data & Results: Test °F Sampl	3pt, 4:1 span (n=5), 0.05"/min	v Norm. U	LR# 28134 Checked by: nit N-% Rprt #
FV 0.5"x2.0" (n=3) TEST FV/VV/PPT (n=3) 0° SBS per ASTM D 2344-84 @RTD, sting Data & Results: Test °F Sampl	3pt, 4:1 span (n=5), 0.05"/min	v Norm. U	LR# 28134 Checked by: nit N-% Rprt #

#### Test Results

Form Rev: 3/1/10- BRM

# Appendix D

	<ul> <li>In-process</li> </ul>	Sec. Sec.	
P/O #	_	LR # 2813	35
Index#	— WO #	Date 1/16	5/2019
Need Date		Start Date 01/	16/2019 -
Customer TCAC		Test End Date	
Test Spec. TCAC Internal	Cert. #	Test Time	737075
Description TC1000 PEI, AS4 145 gsm	32%RC TCAC# 62124582	2 Type Tape C	ure Thermoplastic
Fiber/Fabric AS4 Area	Wt. 145 Resin PEI	Width 11 R	C% 32
Sample Requirements	Box#	TAR# Test L	ead Time
al Poly Senior Project	Material Lot # 021715.1TD		ook Cada
	Material Lot # 051/15-11P.	FIG	ect code
	Resin Lot # TBD	T	est Code Other
e-Test Preparation & Inst	ruction:	-	
AT-UP OVAV		APRT	106491
		1100	1.0001
[1 - 28 ply]6"x6" [0/90]7s ] - 6"x	6" (make panel to fit frame and to	thickness minimal over)	000488
CURE			
		1	
Press process in picture frame, (2	2 bar 10°C/min. to 330°C, 30 bar	, 60 min. and 10°C/min. cool	)
		~~~	
MACHINE		NR .	
		767rd	
SBS per ASTM D2344		10 207	
100 per A0111 02011			
2/05/20//(2-2)			
v 0.3 X2.0 (II-3)			
TECT			
EST			
-v/vv/PP1 (n=3)			
)° SBS per ASTM D 2344-84 @R1	TD, 3pt, 4:1 span (n=5), 0.05"/m	lin	
sting Data & Recults.			104 20125
sting bata a results.		Checke	ed by:
Test °F Sar	nple Information Lot#	Raw Norm. Unit N-	% Rprt #
and she had			
it Results		Fo	rm Rev: 3/1/10- B