ME533 – Applicability of the Composite Rule-of-Mixtures for the Description of the Mechanical Properties of PBT Polymeric Matrix-Short Glass Fiber System

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1 Introduction

This lab is meant to demonstrate the applicability of the composite rule of mixtures to describe the mechanical properties of PBT polymeric matrix, short glass fiber systems.

2 Experimental Procedure [1]

For this lab, samples of PBT11, PBT12, and PBT14 were supplied. For instructions on how to prepare these samples, refer to the lab manual [1].

There were two major steps in performing this experiment: polishing and microscopy.

2.1 Polishing

1. Beginning with 240 grit wet sandpaper (hand grinder), grind the specimen in one direction until all scratch marks line up in the grinding direction. Move up to the next grit of sandpaper
2. Rotate the specimen 90° to the scratch marks and grind until the scratch marks are removed.
3. Repeat steps 1 and 2 until all grits of sandpaper are used (240, 320, 400 and 600).
4. Use the knuth rotor and gently place the specimen on the 1000 grit sandpaper wheel at 90° to the previous scratch marks.
5. Proceed to the polishing wheel and install the $1\mu m$ polishing wheel. Turn it on and pour $1\mu m$ alumina powder mixture on the wheel. Again, gently place the specimen on the wheel at 90° to the previous scratch marks
6. Repeat step 5 using finer and finer alumina powder ($0.3\mu m$, $0.1\mu m$, $0.05\mu m$). Clean the wheel of any residual powder after each polishing using water and a hand brush.
7. Clean the sample with water and place it in the ultrasonic cleaner.
8. Clean the sample with alcohol using a cotton ball and then air dry.

2.2 Microscopy

1. Using Image Pro Plus, focus the sample under the microscope and adjust the lighting and zoom, as needed.
2. Click *Capture* in the top left menu to capture the image. The image will be saved at the bottom of the page. Click on the image to open it.
3. To add scale lines, click on *Measure* and then *show scale line*. Click on *select* and drag the scale marker to the desired location.
4. Double click on the scale marker to adjust the scaling as needed.
5. Click on *calibration table* and select the zoom used when taking the image and apply it to the image.

2.2.1 Measuring Phase Fraction

6. For the transverse view of the sample, measure the field of view area. Click *insert rectangle* and drag it over the whole frame and double click it.
7. Measure the area of the fibers. Click *2 point circle* and create circles around all the visible fiber cross-sections.
8. Measured values will show in *measurement list*.
9. Find $V_f = A_{fibers}/A_{image}$.

2.2.2 Measuring Distance

10. For the longitudinal view of the sample, measure fully revealed fiber lengths. Click on *line* and create a single line per fiber, dragging it from start point to end point.
11. Measured values will show in *measurement list*

3 Results

From conducting the experiment and examining the microstructures of each of the compositions (PBT12, PBT11, and PBT14), the following microstructural properties can be found. Since PBT14 had no fibers, these values are not applicable for it.

- Average volume fraction of reinforcing fibers
- Average length of the fibers
- Average diameter of the fibers
- Average aspect ratio
- Volume fraction of matrix
- Volume fraction of voids (qualitative)

These values were determined by counting and measuring fiber lengths and diameters seen under the microscope and averaging the greatest 20 results. Figures 1, 2, 3, and 4 show how this was done. In both samples, the fibers were counted and measured in various places around the sample area. For the longitudinal fibers, only fully revealed fibers were counted (rectangular ends). The volume fractions for the samples of PBT11 and PBT12 were calculated by determining the ratio of the cross-sectional area of the transverse fibers in the field of view of the microscope to the field of view of the microscope.

Figure 1: PBT11 Longitudinal Fiber Counting

Figure 2: PBT11 Transverse Fiber Counting

Figure 3: PBT12 Longitudinal Fiber Counting

Figure 4: PBT12 Transverse Fiber Counting

The following table lists the respective values for PBT9, PBT11, PBT12, and PBT14. For PBT9 it was assumed that the fibers used in the composite would be the same as PBT11 and PBT12. The fiber lengths and diameters were simply calculated as the respective average of the same values for PBT11 and PBT12. Since PBT14 had no fibers in the polymer, these values were ignored, or set to zero.

Table 1: PBT9, PBT11, PBT12, and PBT14 Values

Sample	PBT9	PBT11	PBT12	PBT14
V_f	0.224	0.179	0.135	0
$\ell(\mu m)$	294.14	299.33	288.94	N/A
$d(\mu m)$	14.61	14.60	14.61	N/A
ℓ/d	20.14	20.50	19.77	N/A
V_m	0.78	0.82	0.86	1
V_v	Less	Less	Many	Many

The following figures show tensile tests performed for samples of PBT9, PBT11, PBT12, and PBT14 in the elastic regions and up to fracture.

Figure 5: PBT9 Tensile Test

Figure 6: PBT9 Test to Failure

Figure 7: PBT11 Tensile Test

Figure 8: PBT11 Test to Failure

Figure 9: PBT12 Tensile Test

Figure 10: PBT12 Test to Failure

Figure 11: PBT14 Tensile Test

Figure 12: PBT14 Test to Failure

Observing these graphs, it is clear that the modulus is fairly consistent for each of the PBT samples, but when it comes to the fracture point, the test samples performed slightly differently. For PBT9, the fracture strength ranged from 97MPa up to 112MPa, with a strain at break of 4% to 6%. For PBT11, the data is more precise, with the fracture strength ranging from 110MPa to 113MPa, with a strain at break of 5% to 6%. For PBT12, the data is also fairly precise, with the fracture strength ranging from 60MPa to 67MPa, with a strain at break of 4.5% to 5%. Finally, PBT14 had precise fracture strengths (31MPa to 34MPa), but very diverse strains at break (40% to 130%).

Table 2 shows the mechanical material properties of the fiber (E-Glass) and matrix (PBT) materials. These will be used later on when calculating the Young's Modulus and Fracture Stress of the samples. The Young's Modulus for PBT is determined from Figure 11 and the fracture strength and yield stress for PBT is obtained from observing the yield points of Figure 12.

Table 2: Material Data for E-Glass Fiber and PBT Polymer

Material	Young's Modulus (MPa)	σ^*, Fracture Strength (MPa)	Shear Modulus (MPa)	Yield Stress (MPa)
$E - Glass$	70000	1750	881.6	N/A
PBT	2558	33.65	N/A	60.91

4 Discussion

Using the values in Table 1 and Table 2 the elastic modulus of the samples can be found using both the Halpin approach (Equation 2) and simple equation (Equation 5). First, it is helpful to determine which set of equations are valid for this case. We assume for these calculations that the fibers are short, discontinuous and parallel to the load direction as seen through the pictures on the microscope. From this we can select the equations which are presented below. These equations require the *critical fiber length*, ℓ_c, which is minimum length of fiber that is required to build up tensile stresses up in the fiber to the fracture strength. This is calculated using the following equation

$$\ell_c = \frac{\sigma_f^* r}{\tau_i^*} \tag{1}$$

The interfacial shear strength, τ_i^*, is found to be equal to the shear yield of the matrix, τ_m^y, which is determined by

$$\tau_i^* = \tau_m^y = 0.5\sigma_m^y \tag{2}$$

The value for the yield strength for the matrix, as mentioned prior, can be found from 2 to be 60.91 MPa.

$$\frac{E_{\downarrow\downarrow}^{short}}{E_m} = \frac{1 + \xi\eta V_f}{1 - \eta V_f} \tag{3}$$

To calculate $E_{\downarrow\downarrow}^{short}$ using the Halpin method, two variables are required, η and ξ, which are calculated using the following:

$$\eta = \frac{E_f/E_m - 1}{E_f/E_m + \xi} \tag{4}$$

$$\xi = 2\frac{l}{d} + 40V_f^{10} \tag{5}$$

The values for the Young's Modulus of the fiber and the matrix can be found in Table 2 and the ratio of length, ℓ, over diameter, d is the aspect ratio which is determined from the results of measuring the fibers and diameters from Table 1.
The simple method of calculating $E_{\downarrow\downarrow}^{short}$ is shown in the following equation.

$$E_{\downarrow\downarrow}^{short} = \eta_l E_f V_f + E_m(1 - V_f) \tag{6}$$

The simple method is developed from the rule of mixtures for a unidirectional lamina and applying a length correction factor, η_l which is found using the following equations.

$$\eta_l = 1 - \left[\frac{\tanh 0.5\beta l}{0.5\beta l}\right] \tag{7}$$

$$\beta = \left[\frac{2G_m}{E_f r^2 ln(\frac{R}{r})}\right]^{1/2} \tag{8}$$

The shear modulus for the matrix can be found in Table 2. The ratio of interfiber spacing, R and fiber radius, r can be found using the following equation from Hull [2]. This relationship is for hexagonally arranged fibers but because the ratio of $\frac{R}{r}$ appearing in a logarithmic term in β , the hexagonal arrangement is acceptable to use as the result of the logarithmic term is sufficiently insensitive to fiber arrangement.

$$\frac{1}{V_f} = \left[\frac{R}{r}\right]^2 \tag{9}$$

The *critical fiber length* for the PBT samples are approximately 815 microns, and since the average fiber lengths found in the samples (Table 1) are lower than the critical length, it is valid to use following equation for fractures stress of the composite. The values for the fiber and matrix fracture strength can be found in Table 2.

$$\sigma^*_{\downarrow\downarrow} = \left(\frac{l}{2l_c}\right)\sigma^*_f V_f + \sigma^*_m V_m \tag{10}$$

The results of these calculations are shown in the following table.

Table 3: PBT9, PBT11, PBT12, and PBT14 Modulus and Fracture Stress

Sample	PBT9	PBT11	PBT12	PBT14
$E^{short}_{\downarrow\downarrow}$ Halpin (MPa)	12658.3	10508.06	8376.91	2558
$E^{short}_{\downarrow\downarrow}$ Simple (MPa)	12256.6	10366.57	8338.3	2558
$\sigma^*_{\downarrow\downarrow}$ (MPa)	163.50	139.19	110.30	33.65
$\ell_c(\mu m)$	815.28	814.93	815.64	N/A
Halpin vs. Simple, % Difference	6.07	5.12	3.18	0

Comparing the results of these tensile tests to the analytical Halpin data in Table 3, it is clear that there is some degree of error between the experimental and analytical results for PBT11 and PBT12. It was previously when doing the experiment that the tensile test for PBT12 was done incorrectly, leading to erroneous values for determining the Young's Modulus.

Table 4: Tensile Test Data

Sample	Young's Modulus (MPa)			Fracture Stress (MPa)			V_f
	Test	Calculated	% Error	Test	Calculated	% Error	
PBT9	13448	12658.33	6.24	107.01	163.50	34.55	0.224
PBT11	10180.33	10508.06	3.12	111.94	139.19	19.58	0.179
PBT12	11957	8376.91	42.74	65.31	110.30	40.53	0.135
PBT14	2558	2558	0	33.65	33.65	0	0

Plotting the experimental and analytical values of the Young's Modulus and the Fracture Stress helps to exemplify the differences between the experimental and analytical results, as shown in Figures 13 and 14.

Figure 13: Fracture Stress, Experimental vs. Analytical

Figure 14: Young's Modulus, Experimental vs. Analytical

As mentioned before the test for PBT12 lead to erroneous results for the experimental Young's Modulus value. Therefore, PBT12 has the largest error of 42.74%. The rest of the analytical values for PBT9 and PBT11 are within a 6% error of the experimental values. It can be seen from Figure 13 that as you go from PBT14 which contains no fibers to PBT12, 11 and 9, the Young's Modulus increases. This is attributed to the increasing volume fraction of E-Glass fibers. This can be predicted from the relationship in Equation 5 for the calculation of Young's Modulus using the simple, Rule of Mixtures (ROM) approach. The experimental values should also show the same trend as the analytical values, but again, because of the error with testing PBT12, it shows a higher Young's Modulus than that of PBT11 which is not to be expected. This trend is plotted in Figure 13.

When calculating the Young's Modulus, there is some discrepancy between the Halpin approach and the simple approach. Both approaches try to use the distribution and geometry of fibers to determine the overall Young's Modulus of the resulting composite. The ROM method utilises a correction factor, η_l, based on the fiber packing geometry to estimate the microstructure of the composite while the Halpin approach uses the aspect ratio, $\frac{\ell}{d}$. The calculated values for both as displayed in Table 3 show that the simple method has a slightly larger value than the Halpin method by up to a 6% difference but have the same trend as shown in Figure 14.

In terms of fracture strength the difference between the analytical and experimental values is up to 40% higher which is a significant difference from the experimental value. This results in a steeper trend line for the analytical values for fracture strength. There is a discrepancy in the trend of the experimental data. As seen in fracture strength values in

Figure 8, the experimental data for PBT11 displays a higher fracture strength than that of PBT9. From the relationship given by Equation 8 and the trendline for the experimental values in Figure 8, the trend should be linearly increasing fracture strength with increasing volume fraction of fiber.

5 Conclusion

We did some stuff using some PBT samples for the sake of testing some stuff. From testing and analysis we learned that something about the PBT or composites and we got these results. From this we can say that blah blah blah or something.

References

[1] R.A. Varin, *"ME533 – Composite Materials Laboratory Research Project*, 2017

[2] D. Hull, *"An introduction to composite materials"*, Cambridge University Press, 1981