

Mechanical Behavior of Compression Molded Polyethylene Terephthalate

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Abstract

The purpose of this project was to investigate the effects of time, temperature, and pressure on the tensile strength and elastic modulus of recycled PET. Compression molding trials were performed on shredded PET bottles to produce tensile test specimens conforming to ASTM D638-03. Aluminum molds containing the PET were mechanically fastened together at the desired pressure and heated in an electric oven. The resulting specimens were subjected to tensile testing for analysis. This screening experiment failed to generate any statistically significant data concerning the factors of interest. These preliminary results may be used to design a more systematic follow-up study.

Introduction

Initially, the intent of the project was to design and construct a hot press capable of compression molding polyethylene terephthalate (PET) bottles as a method of reducing waste and producing value-added goods in developing nations. However, it was quickly realized that the initial goals were overly ambitious and needed to be revised. After rethinking the project, the goal became to design and construct a device to compression mold shredded PET bottles and produce tensile test specimens to measure the changes in mechanical properties, specifically the changes in tensile strength and elastic modulus. The experiment of compression molding was also meant to identify and optimize any significant molding parameters to achieve the greatest mechanical properties. It was believed that information from these tests would allow future work to be done on designing methods to manufacture products from PET bottles.

Polyethylene Terephthalate: Structure and Crystallization

The PET polymer is composed of an aromatic ring and two ester groups at the ends of the molecule (Figure 1). The aromatic ring improves the stiffness of the polymer, particularly in the crystalline regions where the PET chains are aligned. Crystalline PET forms a triclinic semi-crystalline structure that increases the strength and affects the optical properties of the polymer.

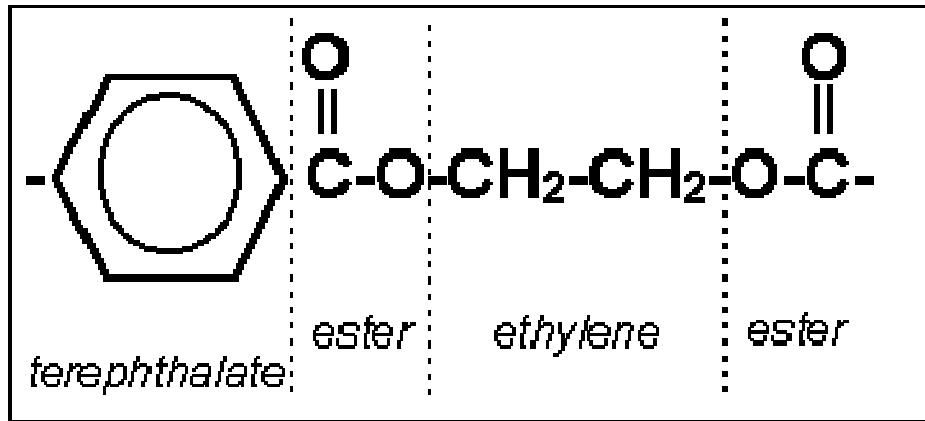


Figure 1: Polyethylene Terephthalate molecular structure. The presence of the aromatic ring in the structure of PET increases its strength and stiffness. The Ester groups serve as points to join monomers into long chains.¹ Image Credit: Kenplas Industries Limited².

When PET crystallizes during cooling, the chains align into lamellae forming spherulitic structures (Figure 2). In semicrystalline PET, the spherulites act as a precipitate surrounded by the amorphous polymer.

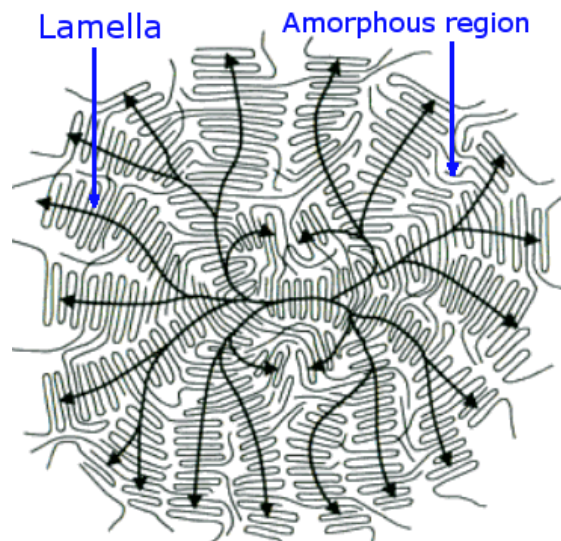


Figure 2: Schematic diagram of polymer spherulites. During crystallization, the lamella radiate outward from a central nucleation site.³ Image Credit: Wikipedia.

Crystallization in PET can be induced by either mechanical stress or thermal energy. Stress induced crystallization occurs when heated PET is strained, causing the alignment of the polymer chains. Stress induced crystallization of PET has been observed when PET was loaded in compression, particularly at strain rates of about 10^3 s^{-1} and strains greater than 140%⁴. At temperatures between the glass transition and the melting temperature of PET, slight crystallization can be induced by pressing the mold dies together, leading to slightly improved mechanical properties and an opaque appearance. The thermal properties of PET are outlined in Table I. Thermally induced crystallization occurs when PET heated above the glass transition temperature cools slowly to allow the nucleation and growth of a spherulitic semicrystalline structure. Amorphous PET can be produced by quenching from the melt. By quenching the PET, the polymer chains are not allowed sufficient time to nucleate and grow spherulites at elevated temperatures.

Table I: Thermal Properties of PET ⁵

Glass Transition (°C)	Melting Point (°C)	Specific Heat (kJ/kgK)
67 - 81	250	1.0

Crystalline polymers have heterogeneous structures with interspersed crystalline and amorphous regions. The degree of crystallinity in polymers is dependent on several intrinsic factors including the distribution of molecular weights, average molecular weight, and extrinsic factors such as the temperature of crystallization, the mode of extension and the stretch ratio. The degree of crystallinity of polymers affects the tensile strength, yield strength, elastic modulus and the glass transition temperature.

Semicrystalline PET also tends to have an opaque appearance whereas

amorphous PET is optically clear. Typical mechanical properties for PET are listed in Table II. Amorphous PET has a glass transition of about 67 °C and crystalline PET has a glass transition of about 81 °C. PET is a hard, stiff and strong material with a decent resistance to degradation upon exposure to chemicals and absorbs little water.³

Table II: Mechanical Properties of PET ⁵

Tensile Strength (MPa)	Elastic Modulus (GPa)	Hardness (Rockwell)
80	2-4	M94-101

Polyethylene Terephthalate: Degradation

PET can degrade as a result of a variety of mechanisms, most notably by hydrolytic and oxidative mechanisms. Hydrolytic degradation occurs when PET is elevated to temperatures above 160 °C in a wet environment, or with moisture present in the polymer. Since PET is able to absorb as much as 0.6% water, it is necessary to dry PET in an atmosphere with a dew point below -10 °C before processing at elevated temperatures to prevent hydrolytic degradation. This can be accomplished by drying in a vacuum oven slightly below 160 °C for about four hours. Oxidative degradation occurs when PET reacts with atmospheric oxygen. Oxidative degradation begins to occur at temperatures above 200 °C and becomes significant at about 300 °C.⁶ This form of degradation can be prevented by processing PET in a dry nitrogen atmosphere. Both hydrolytic and oxidative degradation cause discoloration, a reduction in molecular weight, and an increase in carboxyl end group concentration. Hydrolytic degradation can also cause a reduction in sample mass. Reductions in molecular weight resulting from degradation adversely affect the mechanical properties of PET.

Polyethylene Terephthalate: Recycling

The traditional recycling process begins with the collection of recyclables, which are then transported to a recycling facility and sorted according to material type. PET is identified by the recycling code 1, and is separated from all other plastics. Next, the bottles are washed and then chopped in an industrial shredder. Once out of the shredder, the flakes of PET are thoroughly washed and dried, packaged, and shipped off to outside manufacturing facilities where they are recycled into new products.

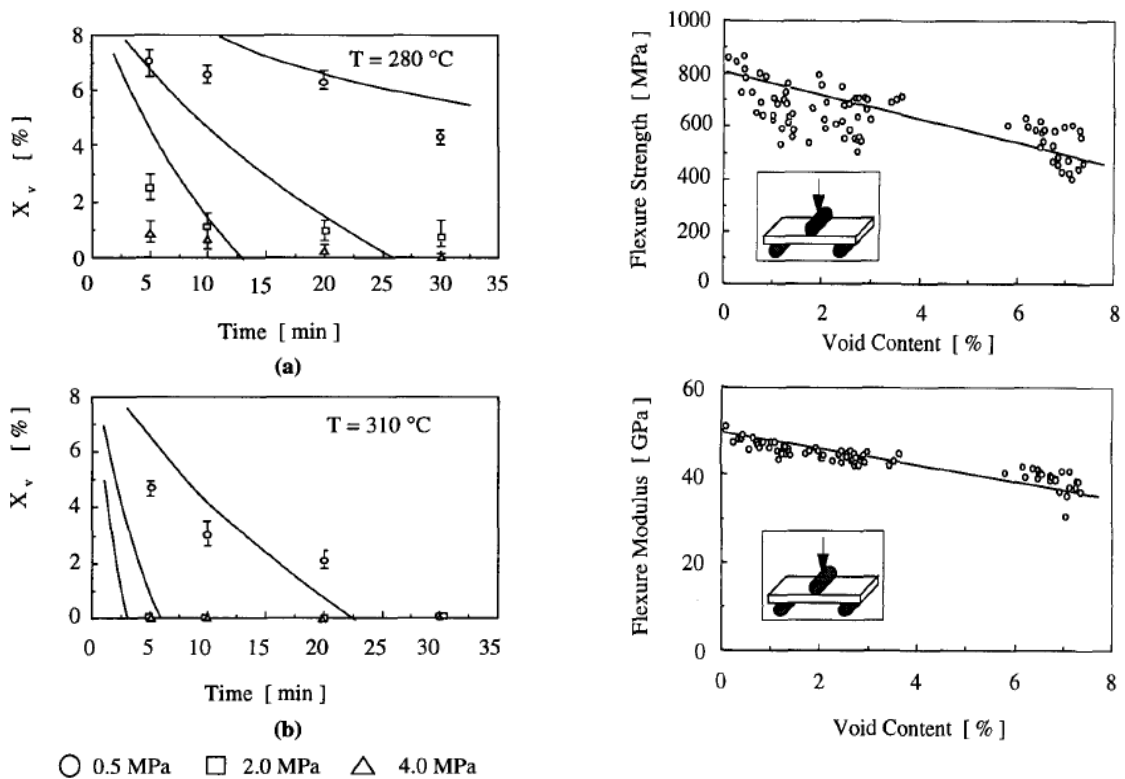
Compression Molding

Compression molding was originally used in the baking industry to shape cookies. In the early 1900's, a technique was developed to mold Bakelite compounds. Then the method was adopted by the plastics industry early on to shape plastic parts such as buttons, dishware, and appliance housings⁷. Today, compression molding is most often used in the processing of thermoset matrix composites such as sheet molding compound (SMC), bulk molding compound, and thermoplastic matrix composites such as long fiber reinforced thermoplastics and glass fiber mat reinforced thermoplastics⁷. Compression molding employs high pressures and temperatures to remove voids, improving the mechanical properties of the resulting composite material.

In compression molding, the raw material is placed between two heated dies in the open position. Once loaded, the dies are closed, forcing the material to flow into the void between the cavity and the core. The mold is closed under pressure for a specific time depending on the material to cure the material into the shape of the mold. The

product is then removed from the mold cavity for inspection. Compression molding can only make simple parts since hollow areas cannot be made and thin areas are hard to fill. Compression molding doesn't require runners or gates for the material to flow through, so composites with fiber reinforcements can be molded to achieve high strength properties. When thermoplastics are used, often the prepreg is pre-cut into a desired shape to save on material costs⁷. Compression molding of thermoplastics reduce the production rate because the polymer requires longer heating and cooling times. The mold is generally made from aluminum, cast iron, or steel. It is heated to the curing temperature (typically 140-160°C) by steam or electricity⁸.

Properties of a product from compression molding vary depending on the time, temperature, pressure, and material. The consolidation quality of the product greatly affects the mechanical properties. In a study conducted by the Cooperative Research Centre for Aerospace Structures, both the flexural strength and elastic constant of the composite was dependent on the consolidation quality (Figure 3). The consolidation quality is determined from the void content in the material. It was determined that higher temperatures, pressure, and longer times would decrease the void content, resulting in a higher consolidation quality (Figure 4)⁹.



Figures 3 & 4: (3, left) Mechanical properties of carbon fiber/polyetherimide composite as a function of void content⁹ and (4, right) Void content as a function of time, temperature, and pressure.⁹

Current technology involving blow molding of PET is almost entirely limited the bottle industry. The pre-forms that are blow molded to create PET bottles have traditionally been injection molded, but the Sacmi Group, an Italian company that manufactures the machines that are used for PET bottle production, has begun to explore compression molding technology. Compression molding offers greater efficiency, production quality, and cost savings when compared to injection molding¹⁰.

Studies involving compression molding of thermoplastic SMC composites assisted in predicting the behavior of PET during molding. When compression molding SMC, plies are first cut from the SMC sheet. They are then stacked to make a charge and placed into the bottom of the mold that has been heated to about $150\text{ }^\circ\text{C}$ for 5-20

seconds. This allows the SMC to preheat before the mold is closed. When preheating PET, the temperature has not reached the glass transition temperature of PET, so the polymer chains are still rigid. As the temperature increases and reaches the glass transition state, the chains become more flexible and can unfold under stress. Then the mold is closed to squeeze the charge, deforming the SMC, and reducing the height of the charge. Under compression, while the chains are flexible, the PET is able to randomly coil and entangle. The chains straighten and some even slide over to their near neighboring chain. Here, the chains are parallel and are closely packed. When the mold is filled, it is kept closed for a curing stage for 1-2 minutes¹. For thermoplastics such as PET, a curing stage is unnecessary. The product is then removed from the mold. If the PET is not quenched, the polymer has an opaque color from the spherulitic structure by thermal crystallization aggregates that are incoherent. If the PET is quenched, it forms an amorphous structure.

Materials and Methods

Mold Design and Manufacture

The mold was designed in accordance with the ASTM standard test method for tensile properties of plastics (ASTM D638-03). This standard defines the sample dimensions and operating procedure for measuring the tensile properties of unreinforced and reinforced plastics in the form of tensile bar shapes. It works for testing materials up to a thickness of 0.55". Figure 5 shows a schematic representation of what a tensile test specimen looks like, along with the critical dimensions labeled as variables.

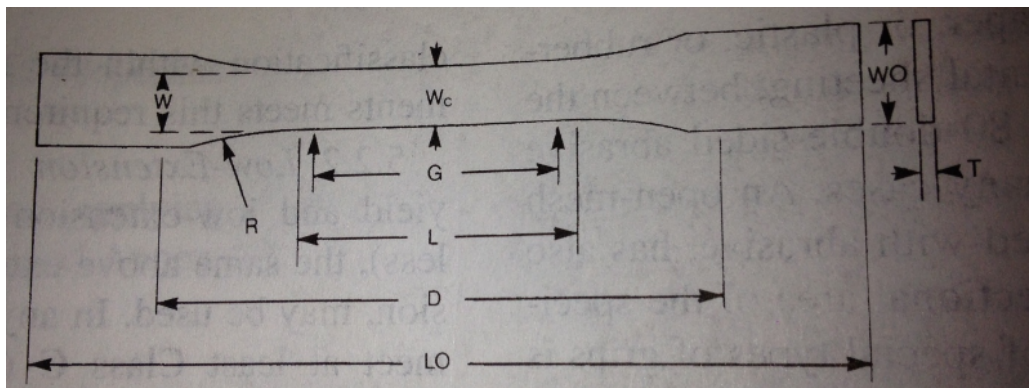


Figure 5: Schematic representation of a tensile test specimen per ASTM D 638-03.

The mold design was designed with PTC Creo software following the Type 1 dimensions in the ASTM standard D 638-03 (Table III). Two 6" x 6" x 0.5" aluminum plates were donated from the Industrial Manufacturing Engineering department to produce the mold. The design and plates were taken to Mustang 60, an on campus machine shop, to be computer numerical control (CNC) milled into the design needed for the mold.

Table III: Tensile Test Sample Dimensions Specified by ASTM D638-03

Dimension Variable and Name	Dimension Size (in.)
W – width of narrow section	0.50
L – length of narrow section	2.25
WO – overall width	0.75
LO – overall length	6.50
G – gage length	2.00
D – distance between grips	4.50
R – radius of fillet	3.00

In order to achieve the pressure needed for the design, six ¼” through holes were drilled into both plates. These holes were necessary for the ¼” bolts to create a clamping force. The bolts were tightened using steel hex nuts and a torque wrench. The two finished aluminum plates with through holes are shown below in Figure 6.

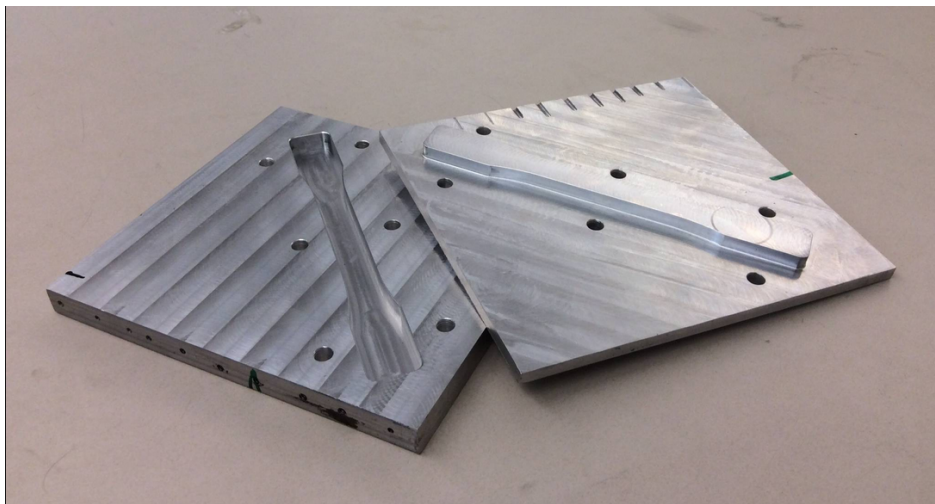


Figure 6: Finished aluminum molds

Sample Preparation

The series of steps taken to prepare the bottles for compression molding closely resembled the recycling process of industrial PET recyclers. First, PET bottles were collected from recycling bins around the Cal Poly campus. The bottles underwent a preliminary rinse to remove residual liquid, then had their labels, tops, and bottoms removed. The PET was then sectioned into large pieces, which were thoroughly hand-washed and dried, then placed in a vacuum oven to complete the drying process. The oven was set to approximately 130 °C for four hours, which ensured water molecules were removed from the polymer chains. Once out of the vacuum oven, the PET was sectioned into thin strips, which were approximately 5" long and varied in width between 1/8" and 1/2". These strips of PET were then arranged into the aluminum mold and heated under pressure. The clamp force applied to the mold by each bolt was estimated by Equation 1:

$$F = \frac{T}{\mu D} \quad (\text{Eq. 1})$$

where F is the clamp force in lbf, T is the applied torque in in-lbs, μ is the coefficient of friction between the bolts and nuts, and D is the nominal (major) diameter of the bolts, measured in inches¹¹.

Test Design

The experiment run on the PET was designed as a screening experiment, the goal of which was to determine which factors, if any, had a significant effect on the mechanical properties (tensile strength and elastic modulus) of the molded PET. Based on background information regarding compression molding, it was determined that the

primary factors of interest would be pressure, temperature, and time. A realistic range of pressure was found to be 125 to 620 psi, which was calculated from the mold geometry and a typical range of torque values (5-25 ft-lbs). In terms of temperature, the PET was subjected to a range between the glass transition temperature and the melting point, which is between 74 and 250 °C. The PET was never subjected to temperatures greater than 250 °C because the experiment was intended to investigate the feasibility of fusing PET below its melting point. The initial time range selected was quite large, between 10 minutes and 2 hours. The upper limit of 2 hours was never exceeded to avoid oxidative degradation to the PET.

Testing Parameters

For the heat treatment of the PET, a fisher scientific low temperature oven was used to create a consistent temperature for the manufacturing trials. During early stages of the trials, it was determined that only the upper levels of the time, pressure, and temperature limits were successful in creating tensile test specimens that showed cohesion between the PET particles and strips.

For the pressure, the 1/4" bolts would break at torque values higher than 12 ft-lbs. It was decided to maximize the torque to 12 ft-lbs for the remainder of the tests. This torque value equated to about 300 psi for 6 bolts, assuming no contact between plate surfaces.

The temperature range was initially 150-250 °C. The early preliminary tests helped determine that temperatures below 200 °C did not show much cohesion between the PET particles and strips. The temperature range was then narrowed down to 200-

250 °C. A temperature above 250 °C was avoided to prevent melting of the PET. During testing, temperature changed in increments of 10 °C.

The time range was initially ten minutes to two hours. Preliminary tests determined that heat treatments below one hour did not create any successful tensile bars with PET that bonded together using torque values of 12 ft-lbs or below. Time above two hours showed discoloration and oxidation, so times above two hours were avoided. Time changed in increments of 15 minutes.

Tensile Testing

An Instron tensile testing machine was used to measure the mechanical properties of each test specimen, specifically its tensile strength and elastic modulus. After taking precise dimensional measurements of the gage length, width, and thickness, they were placed into the testing grips and pulled one at a time until failure occurred.

Differential Scanning Calorimetry

Three DSC tests were conducted using an SII Seiko Exstar 6000. The data was analyzed using the Exstar Analysis software, and exported to Microsoft Excel. The first test was conducted on an as-cut sample from a PET water bottle ($m = 4.8$ mg). The sample was run through two cycles between 20 °C and 310 °C at a scan rate of 10 °C/min. DSC tests were also conducted on samples cut from tensile test specimens #11 ($m = 7.6$ mg) and #20 ($m = 7.1$ mg). In these tests, the samples were cycled twice between 20 °C and 300 °C at a scan rate of 10 °C/min.

Results

Tensile Tests

Out of the 32 trials run, only 20 trials showed cohesion between the PET particles and strips. Of the 20, only 12 were worthy of testing due to delamination between the PET and large voids in the sample. Only the tests with strips of PET were successful in creating the tensile bar specimens with cohesion. The tests with particles of PET quickly crumbled when taken out of the mold. Samples 2 and 20 were the stiffest samples, and Sample 20 was the strongest. Sample 2 was made with thin strips of PET while sample 20 was made with PET laminates cut into the shape of the tensile test specimens and layered.

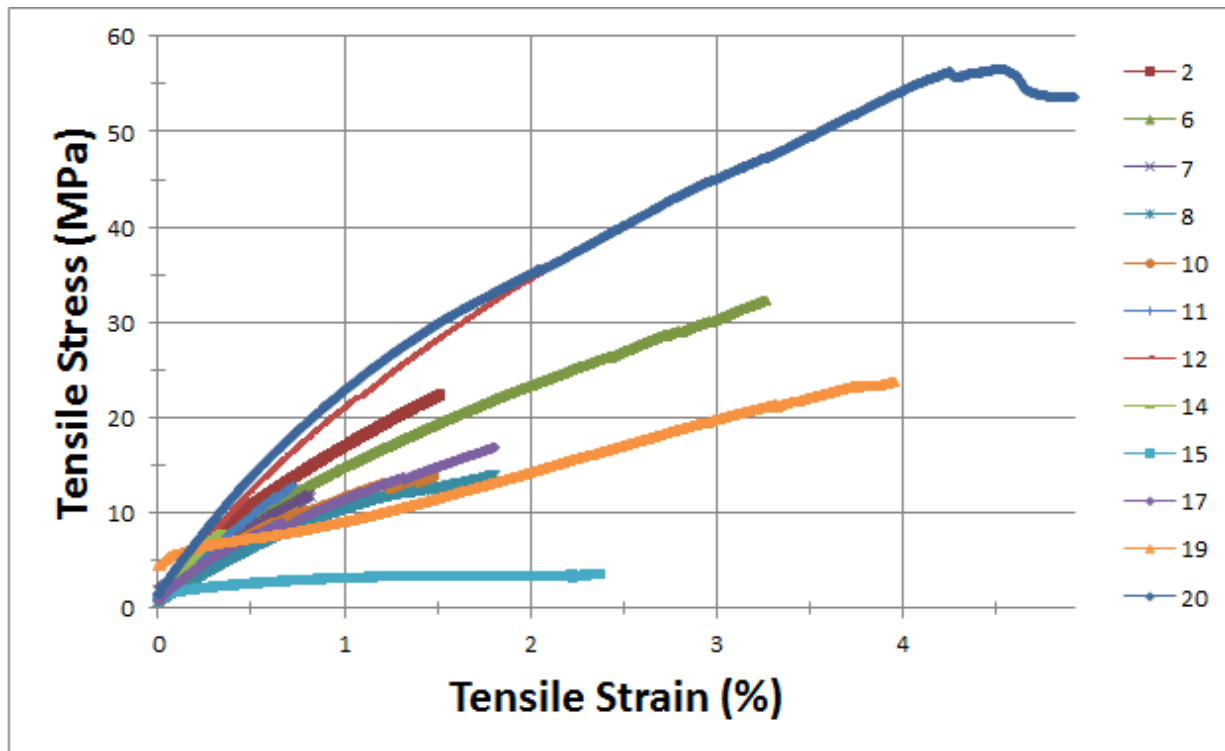


Figure 7: Tensile test results of 12 samples. Samples #2 and #20 showed the most desirable properties.

The sample averages of tensile strength and elastic modulus were significantly lower than virgin PET property values. The tensile strength and elastic modulus of compression molded PET was 72.55% and 52.67% lower, respectively, compared to virgin PET. When comparing the best sample (#20) against virgin PET, the tensile strength and elastic modulus of were only 29.21% and 32.33% lower, respectively. The mechanical property data from the tensile tests is summarized in Table IV, and compared to virgin PET values. The standard deviations of the samples property values were large, meaning there was a large amount of variation in the response data.

Table IV: Comparison of Mechanical Properties Between Samples and Virgin PET

	Tensile Strength (MPa)	Elastic Modulus (GPa)
Average	21.96 (80*)	1.42 (3.00*)
Standard Deviation	15.73	0.64
Minimum	3.56	0.64
Maximum	56.63	2.03

* Virgin PET Values

Each of the factors time and temperature were plotted against each of the response variables, tensile strength and elastic modulus (Figures 8-11). Each of the plots contains red and blue dots, which were used to signify the different-sized test strips used in sample preparation. The blue dots represent the thin strips (approximately 1/8") and the red dots represent the thick strips (approximately 1/2"). Regardless of strip size, the scatterplots all fail to show any noticeable effect of either of the factors on either of the responses. There is a high amount of variability in the response data, which obscures any trends that may be present.

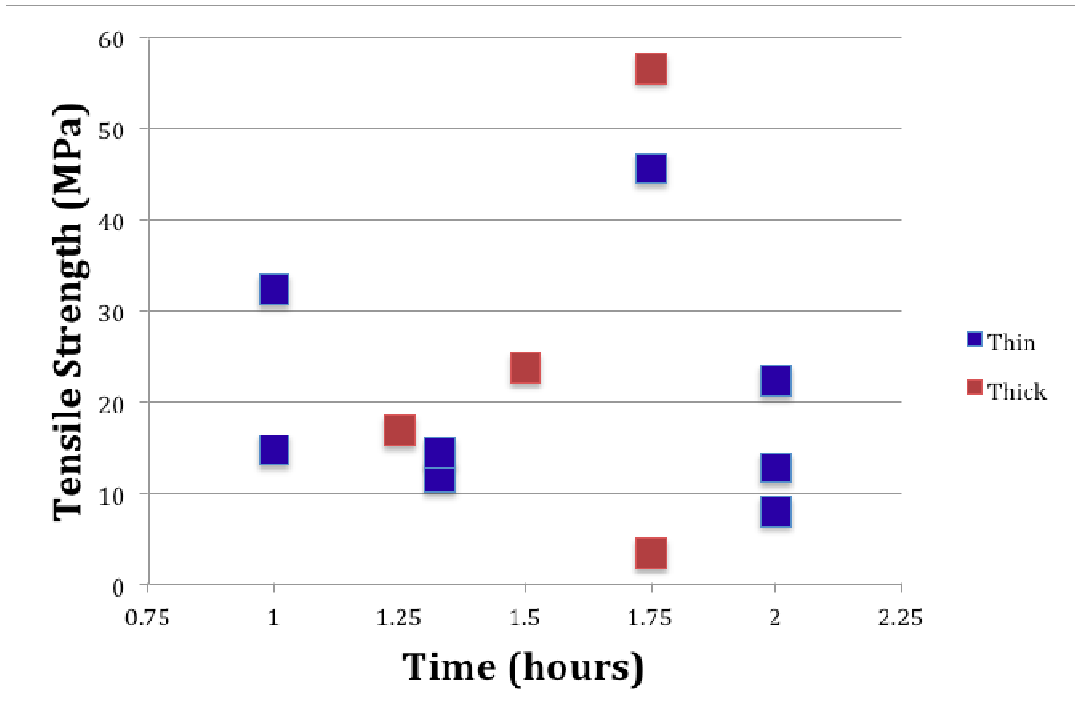


Figure 8: Scatterplot showing the effect of time on tensile strength. Regardless of strip size, there was no clear trend in the data.

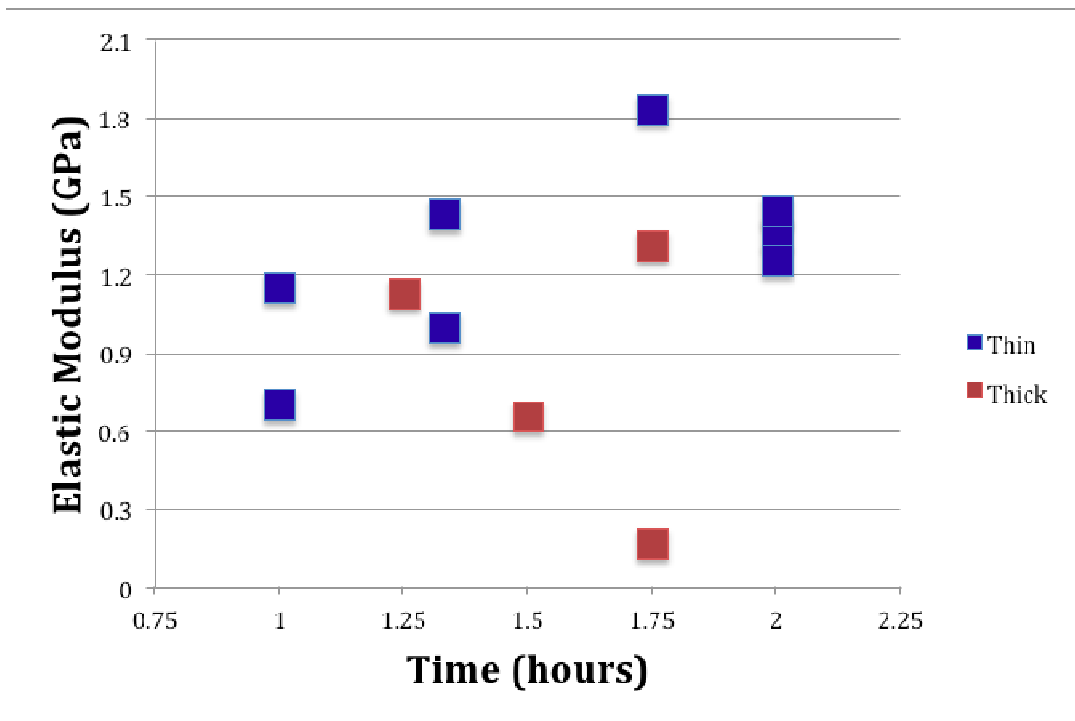


Figure 9: Scatterplot showing the effect of time on elastic modulus. There appears to be a slightly positive effect of time on elastic modulus for the thin strips, but no pattern for the thick strips.

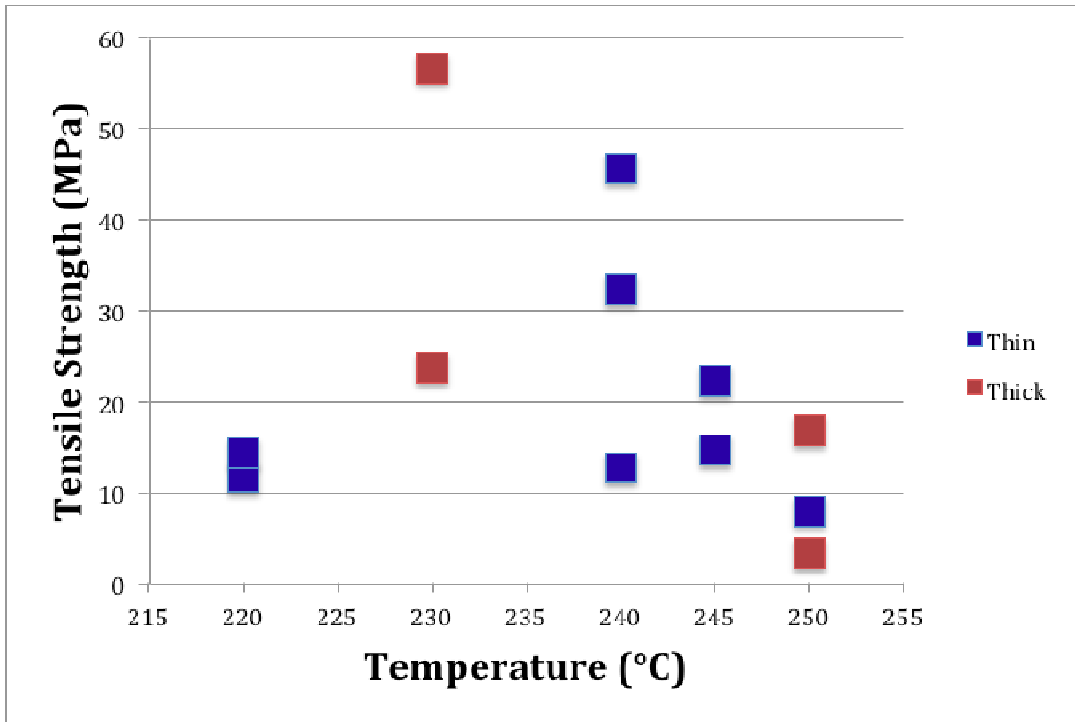


Figure 10: Scatterplot showing the effect of temperature on tensile strength. There was not a visible trend in the data, even after taking strip size into account.

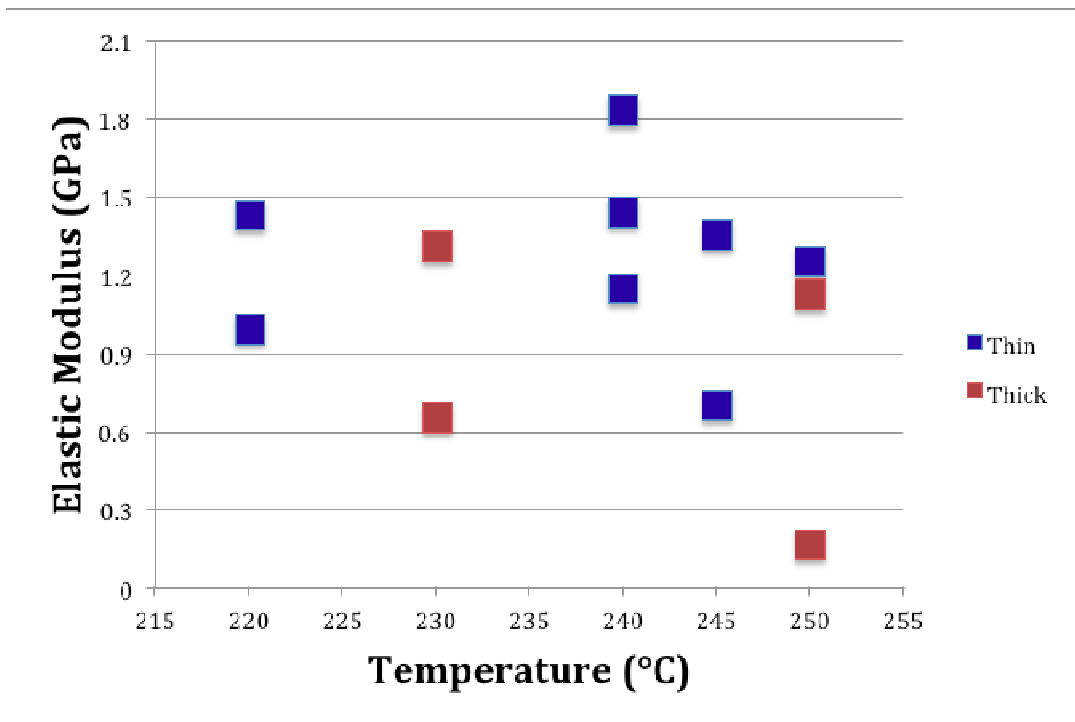


Figure 11: Scatterplot showing the effect of temperature on elastic modulus. There was too much variation in the data to detect a trend for either strip size.

Differential Scanning Calorimetry

Changes in the thermal properties of PET were noticed between the as-cut specimen and the samples tested after molding. Most notably, the as-cut specimen exhibited a cold crystallization peak (Figure 12), and lacked double melt behavior whereas the molded samples did not exhibit the cold crystallization peak, and did exhibit double melt behavior (Figures 13, 14). The phase transitions for each of the samples are provided in Table V.

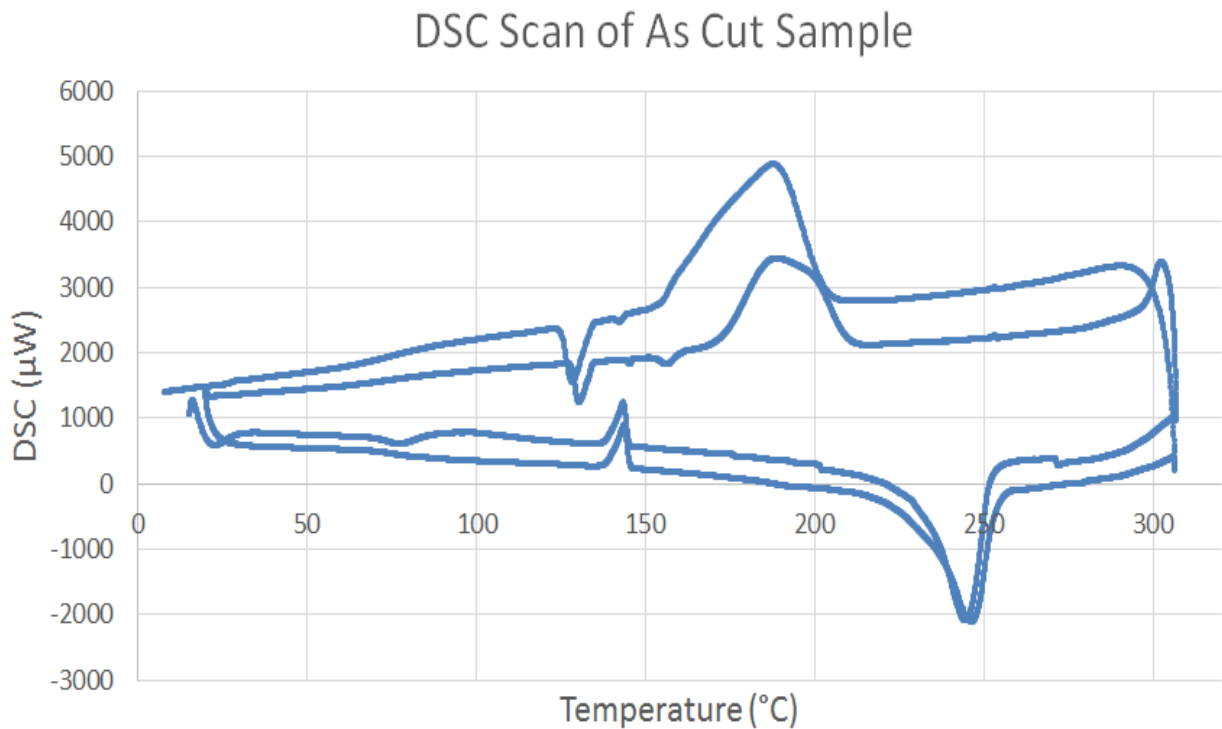


Figure 12: DSC scan of as-cut sample, showing cold crystallization peaks just before 150°C

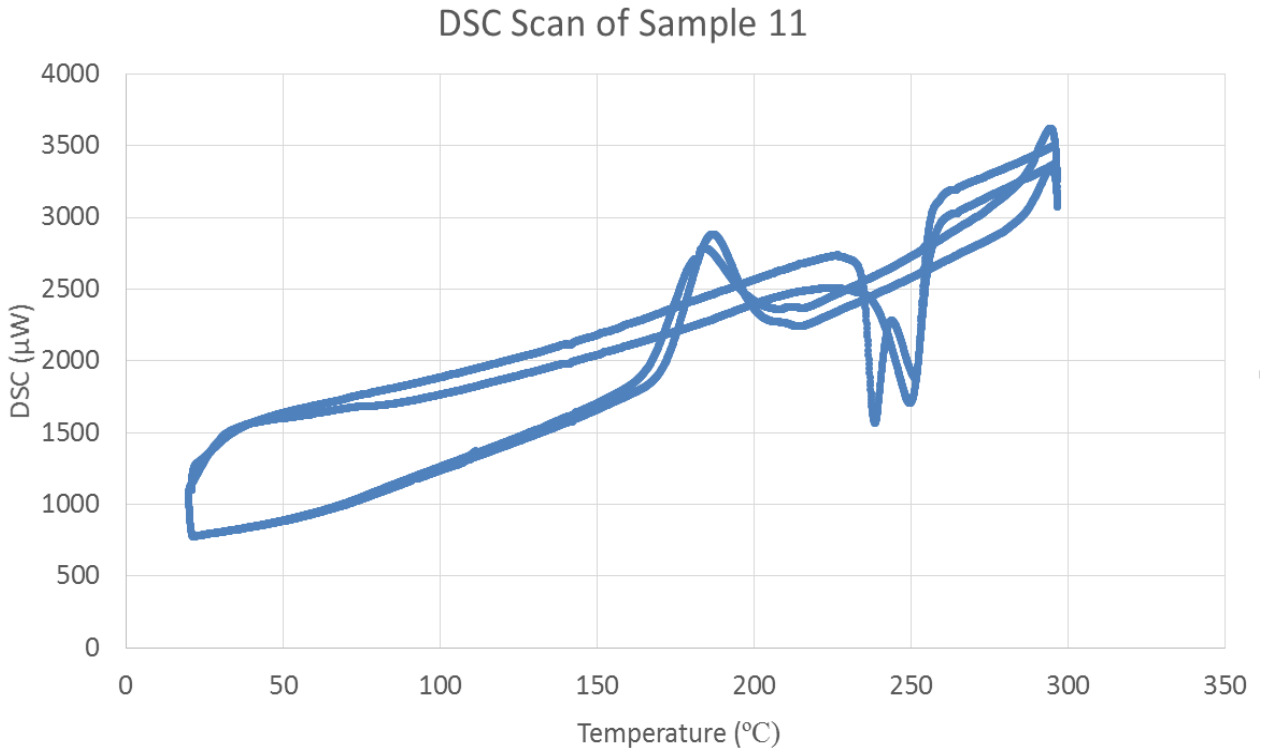


Figure 13: DSC scan of Sample 11, showing double-melt behavior just before and at 250 $^{\circ}\text{C}$.

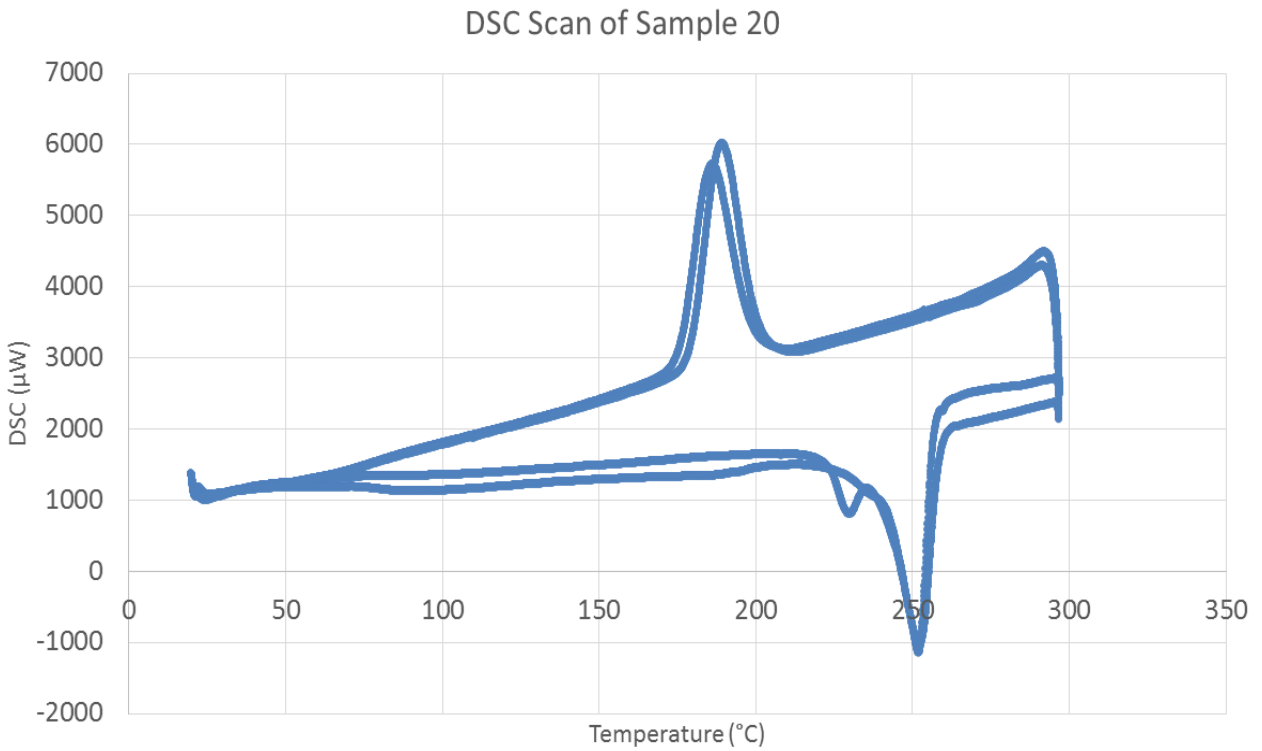


Figure 14: DSC scan of Sample 20, showing double-melt behavior just before and at 250 $^{\circ}\text{C}$.

Table V: Thermal Property Values Obtained from DSC

Properties	As Cut	Sample 11* (cycles not clear)	Sample 20 (cycles not clear)
Glass Transition (Heating)	71.2°C (cycle 1) 72.2°C (cycle 2)	52.9°C, 60.5°C	71.1°C, 74.1°C
Glass Transition (Cooling)	82.6°C (cycle 1) 80.6°C (cycle 2)	Not detected	Not detected
Cold Crystallization Peak	142.6°C (cycle 1) 143.2°C (cycle 2)	Not detected	Not detected
Melt Peak	244.4°C (cycle 1) 246.2°C (cycle 2)	238.6°C (Peak 1) 249.8°C (Peak 2)	229.8°C (Peak 1) 251.6°C, 251.9°C (Peak 2)
Melt Return to Baseline	251.5°C (cycle 1) 253.0°C (cycle 2)	255.4°C, 257.5°C	257.6°C, 259.3°C
Crystallization Onset	209.4°C (cycle 1) 202.5°C (cycle 2)	202.9°C, 203.2°C	199.1°C, 200.7°C
Crystallization Peak	187.8°C (cycle 1) 187.7°C (cycle 2)	183.7°C, 187.6°C	185.9°C, 189.7°C

***Unusual DSC curve indicates possible machine malfunction**

Discussion

After collecting data from the tensile tests, it was determined that a formal statistical analysis would be unjustified, mainly due to the fact that treatments were not randomly assigned to samples. The reason this could not be done was because the sample preparation method was constantly changing and improving over time. With only 12 samples, there was not a sufficiently large sample size to achieve meaningful results. As seen in the scatterplots of time and temperature, there was simply too much variation in the data, so any attempted analysis would yield insignificant results.

Throughout the course of preparing the tensile test specimens, it was noticed that larger pieces of PET improved the form of the molded samples. The shift in preparation method from chopped PET to strips showed the greatest increase in form, and increasing the width of the strips improved the form as well. During tensile testing, the most common method of failure was delamination, as opposed to necking and fracture as expected. It was concluded that it was in fact possible to fuse PET into a shape below its melting point.

Other sources of error during testing include the degradation of the mold as it was thermally cycled throughout the duration of the tests. The aluminum mold exhibited significant deformation when the tests were finished, limiting the pressure that was applied to the specimens, and changing the dimensions of the later samples. The nuts and bolts used throughout testing exhibited surface degradation, increasing surface roughness and likely the coefficient of friction. According to Equation 1, this degradation could have impacted the clamping force applied by each bolt. An additional confounding factor that was not properly controlled for due to time constraints was the method of

sample preparation, which likely varied according to who prepared it. Finally, some variability in the fill of the mold during testing likely impacted the applied pressure. More overfilled molds prevented the mold surfaces from coming into contact, meaning that all of the clamp force was transmitted to the specimen, whereas under filled molds allowed the mold surfaces to contact, reducing the pressure applied to the specimen. The lack of control over these factors was likely responsible for much of the sample variability.

Conclusion & Recommendations

Through our testing, the desired tensile strength and elastic modulus were not achieved by our experimental design. The best samples had about 66% the tensile strength and elastic modulus of virgin PET.

During the term of the project, complications came up which made it hard to make progress with the project and achieve the desired properties. The first complication came from finding literature on compression molding of PET. This made it hard to narrow down the test parameters, so lots of time was spent doing this task. The lack of funding of the project also made it hard to make multiple mold designs to optimize the process. With more funding, more multiple molds could have been made to make the tensile test specimen production much more efficient. The third complication came towards the end of the project. After one of the last tests, it was noticed that the mold was deforming under the dog bone ends. The cause of this was hypothesized to be from the thin mold and the high pressure developed from overstuffing the mold with PET. The mold started to bulge out, deforming the mold and the tensile test specimens.

Future groups who might take up this project would need to make modifications to the procedure used in this project to prevent degradation of their mold as well as sample variability. To prevent creep, it would be necessary to redesign the mold with thicker dimensions or to use a stronger material that is not susceptible to overaging, such as steel. To prevent degradation of the bolt and nut surfaces, it would be necessary to change them more often, so that similar surface roughness could be maintained throughout testing. A major complication faced in this project was the amount of pressure that could be applied to the mold, due to the mold surfaces coming

into contact, resulting in clamping pressures as low as 40 psi. To prevent the surfaces from coming into contact, it would be necessary to design the mold to allow the inner surfaces to come into contact when empty. This would ensure that the outer surfaces of the mold do not come into contact and all of the force was transmitted to the specimen (Figure 15).

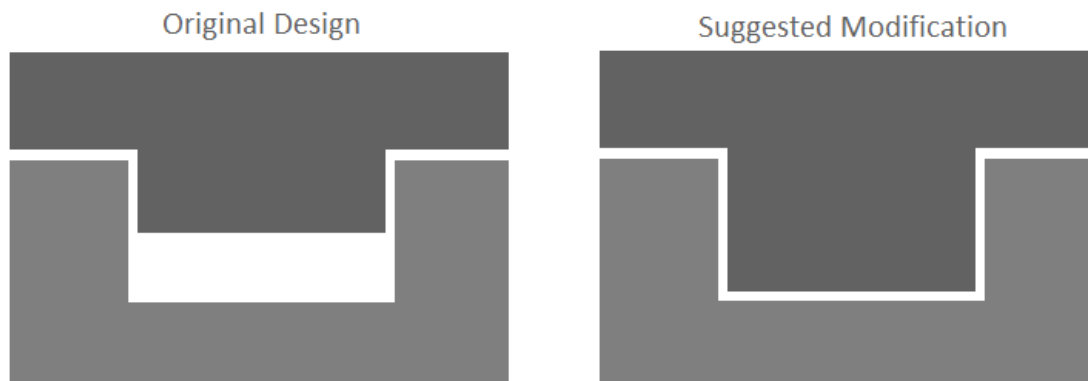


Figure 15: When under filled, the original design allowed the faces of the mold to come into contact, reducing the pressure transmitted to the specimen. Modifying the mold to remove this clearance would allow more consistency in pressure transmission to the specimen at the expense of dimensional consistency.

Another useful modification would be to use thicker bolts, perhaps $\frac{1}{2}$ " diameter. This would allow a higher torque force to be applied to the mold without breaking the bolts. In this project, the $\frac{1}{4}$ "-20 bolts were torqued to 12 ft-lbs (clamp force = 240 lbs), double the rated torque load for SAE Grade 2 bolts. By contrast, $\frac{1}{2}$ "-20 bolts are rated to 63 ft-lbs (clamp force = 504 lbs), and would allow the applied pressure to be doubled¹¹.

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