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Design Process of a Novel Dynamic Mechanical Analysis Sample Preparation Methodology

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The University of Southern Mississippi

DESIGN PROCESS OF A NOVEL DYNAMIC MECHANICAL ANALYSIS SAMPLE
PREPARATION METHODOLOGY

by

Julian M. Richardson

A Thesis
Submitted to the Honors College of
The University of Southern Mississippi
in Partial Fulfillment
of the Requirement for the Degree of
Bachelor of Science
in the Department of Polymer Science and Engineering

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Abstract

The technique of dynamic mechanical analysis (DMA) has been a frontrunner in the field of polymer science for decades. It's used as a tool for characterization has been integral in mankind's understanding of the properties of polymeric materials. The procedure for obtaining data using DMA is widespread and standardized, however the procedure for preparing the samples is a list of dimensions and tolerances with no mention of how to obtain said dimensions. Every research group using DMA is left to their own devices to prepare samples. This discontinuity can create the problem of nonuniform results across the scientific community. This thesis tackles the issue of disjointed DMA sample preparation and investigates a novel method which can be used to standardize and expediate DMA sample preparation in the future.

Keywords: dynamic mechanical analysis, glassy polymers, glass transition temperature

Dedication

To my family, Etharl Richardson and Elisabeth Richardson for your endless love and support, without you all I would never have gotten as far as I have. To the two graduate students, Anthony Benasco and Ashleigh Bristol, that have pushed me and motivated me throughout my undergraduate career. Most, if not all, of my success could be traced back to their support, assistance, and drive. To the undergraduate graduating class of 2018 and 2019, through the countless nights studying together, I have established great friendships and could not have made it through this program without them. Last, but not least, Dr. Morgan for giving me the life-changing opportunity to work in her lab and providing me with many opportunities that would not have been achievable without her.

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List of Abbreviations

| | |
|----------------|-----------------------------------|
| DMA | dynamic mechanical analysis |
| ABS | acrylonitrile-butadiene-styrene |
| T _g | glass transition temperature |
| DSC | differential scanning calorimetry |
| TMA | thermal mechanical analysis |
| PVC | polyvinylchloride |
| TGDDM | tetraglycidyl methylenedianiline |
| 3,3-DDS | 3,3-diamino diphenyl sulfone |
| NPT | national pipe threading |
| GPM | gallons per minute |

CHAPTER ONE: INTRODUCTION

Polymer science has revolutionized the materials market over the past 70 years. Inexpensive, lightweight, and controllable properties are some of the terms one might use when describing polymer products.¹ Although the track record for polymeric materials is impressive, the technology and knowledge that is required to analyze these materials is often complex and, in some areas, rudimentary. Polymers have special properties in that they can be classified as non-Newtonian materials. Non-Newtonian materials have a viscosity that does not change linearly with the rate of deformation or strain.² For melted polymers, this typically means the materials become less viscous with a higher shear rate which is known as shear thinning pictured in Figure 1.³

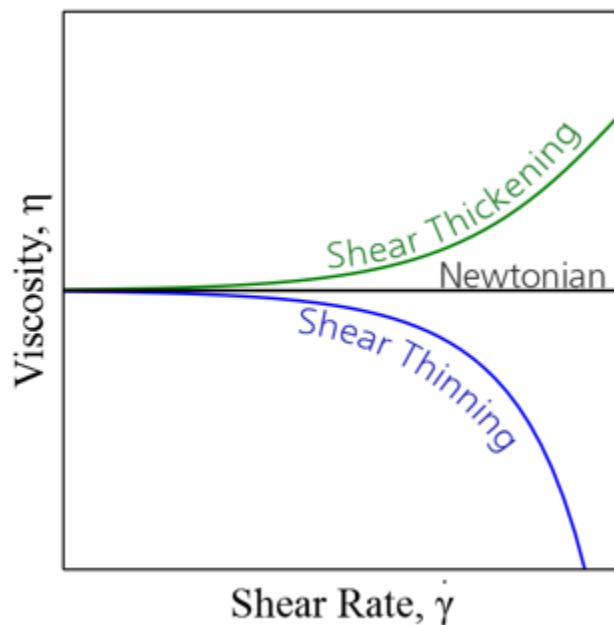


Figure 1, General viscosity vs. shear rate graph for non-Newtonian materials

For solid polymers, materials will exhibit viscoelastic properties. Viscoelastic materials have a combination of viscous properties and elastic properties. This is possible by the molecular interactions between polymer chain side groups. With very high rates of strain, the polymer chains do not have enough time to slip past each other which results in elastic properties becoming predominant. However, with low rates of strain, the polymer chains have time to relax which results in predominantly viscous properties.⁴

The study of the viscoelastic properties in polymers is rheology. Rheology is an indispensable part of polymer property consideration because of the implications it can have on polymer performance. Miscalculations in rheological properties can result in problems such as defective products which can result in consumer injury or death. Currently, Lego® is attempting to overhaul their Lego® bricks from acrylonitrile-butadiene-styrene (ABS) plastic to a more sustainable polyethylene made from plant-based matter.⁵ However, this process will take many years of research. The new plastic that is being considered must be of the same texture, same color, and most importantly have the same distinct clicking mechanism for brick assembly. The mechanism for assembly is dependent on the rheological properties of the polymer. Another important area of concern when working with a new polymer system is finding the appropriate processing conditions that must be used when mass producing the new bricks. Polyethylene will have completely different processing conditions due to rheological flow differences. Any error in calculation can result in large scale defective products that can call into question the integrity of Lego® products. Knowledge of rheology can mean life or death in applications such as aircrafts or space shuttles. Tragedies such as the Challenger incident can occur because of insufficient rheological considerations. One of the main reasons the Challenger exploded was an O-ring that failed because it was too brittle for the application.⁶ Rheology gives useful insights into how a material will behave and that insight should never be overlooked when producing polymeric goods.

A rheological profile of a polymer contains several basic components. Glass transition temperature (T_g), loss modulus, storage modulus, and tan delta are four of the largest factors that dictate how many polymers will physically behave. The glass transition

temperature of a material is the temperature at which a substance transitions from exhibiting glassy properties to displaying rubbery properties. On the molecular level above the glass transition temperature, polymer chains have enough energy and free volume to move past each other and stressors can facilitate segmental chain movements which on a macroscale is the cause of the observed physical properties.⁷ The modulus is an indication of the resistance or susceptibility of a material to deformation when a mechanical stress is applied to the material. The viscous or loss modulus can be described as resistance to deformation that results in heat or “molecular friction.” Deformation energy that results in heat is considered “lost,” ergo loss modulus. Storage modulus is the resistance to deformation that results in potential energy. This potential energy can then be transformed back into kinetic energy after the deformation. Storage modulus is the capability of the material to “spring back.” Tan delta is equal to the loss modulus divided by the storage modulus.

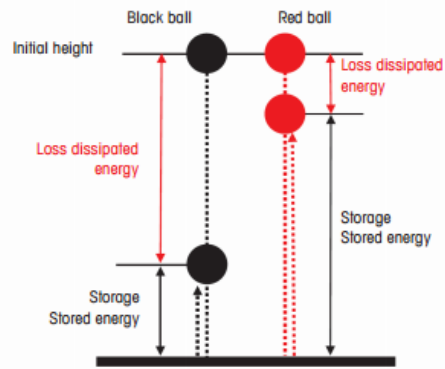


Figure 2, Loss modulus/storage modulus depiction

The greater the tan delta, the greater affinity the material has for releasing deformation energy as heat. The black bouncy ball in Figure 2 is an example of a material with a high tan delta because its loss modulus is larger relative to its storage modulus.⁶ In the case of the red ball, the tan delta is low because the loss modulus is smaller relative to its storage modulus.

Theoretical understanding of the phenomenon of polymer chain movement is useful. However, without a definitive way to measure the rheological properties the theory

could not be applied to the use or design of materials. Several instruments exist which can be used to interpret the rheological properties of polymers. Differential scanning calorimetry (DSC) is one of the fastest ways of measuring a material's glass transition temperature. The instrument works by detecting the heat absorption difference between a reference pan that contains nothing and a sample pan that contains the polymer being investigated. At the glass transition temperature, there is an endothermic peak. This is present because the polymer absorbs energy during the transition. DSCs are readily available in many laboratories, and the sample preparation process is straight forward. However, if a material has a broad glass transition, this technique may not be sensitive enough to detect the transition accurately or, in some cases, at all.⁸

Another technique that is often used to elicit rheological data from polymeric materials is thermal mechanical analysis (TMA). This technique measures the coefficient of thermal expansion of samples as a function of temperature. The instrument holds the sample in place and probes are placed onto the surface of the sample. As the sample is heated and the expansion occurs, the probes move. This movement is measured and can be converted to the coefficient of thermal expansion using known information about the sample. The coefficient of thermal expansion changes dramatically during the glass transition so the glass transition can be easily determined by analyzing the data that is produced. Testing softer amorphous samples can be problematic because they can soften to the point of being punctured by the probes.⁹

One of the most sensitive instruments for glass transition analysis is dynamic mechanical analysis (DMA).¹⁰ The instrument sweeps through a temperature range while the sample oscillates at a fixed frequency to find the temperature/frequency at which the

material resonates. The resonant frequency occurs because the segmental motion of the polymer chains matches the applied frequency and causes the storage modulus to drop significantly at the glass transition, thereby providing a method that can be used to determine the glass transition temperature.¹¹

The standard procedure for using the instrument is straightforward, however, ~~the~~ it is essential to standardize sample preparation procedure to ensure that the quality of polymeric materials is appropriate for the planned purpose, and also that researchers can confidently compare measurements made across different operators, instruments, samples, laboratories, or times. The general sample preparation procedure calls for a small bar of the sample of specific dimensions.¹² However, one would be hard-pressed to find a thorough explanation of the preparation of DMA samples in literature, even though the preparation method may influence the end results of the test. In many research groups that work on high performance polymer networks, the properties of viscoelastic materials such as epoxy amine networks are studied. These materials are very difficult to form into dimensions with specific tolerances on a lab scale. One method used at the University of Southern Mississippi involves mixing an epoxy amine slurry, heating it under vacuum to degas the solution, pouring the hot viscous substance into a preconstructed mold with cavities the size of sample bars needed, then curing it in the oven.¹³ Once this is complete, the sample bars must be sanded down to the tolerances specified

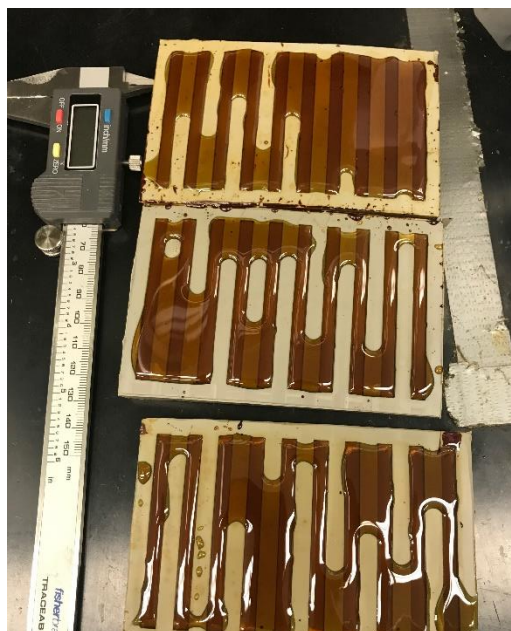


Figure 3, Post-cured DMA bars in silicone molds

by the DMA manufacturer before testing. The molds are typically overfilled for two important reasons: polymer shrinkage may occur during curing which can render samples unusable if the dimensions are under the tolerances allowed for the instrument, and the material is so viscous that it is nearly impossible to correctly fill the cavities with accuracy. Post-cured DMA bars in polysiloxane molds are pictured in Figure 3.

At this point, a problem arises. Sanding down the DMA bars by hand can lead to less than precise thicknesses due to finger divots caused by uneven pressure applied across the bar. Hand sanding is also very labor intensive, has a large learning curve, and can take up to half an hour per bar. This study proposes a solution to this problem, which is faced by laboratories across the country. The proposed design is a tool that holds cured DMA bars in place so an electrical sander can be used to quickly remove excess material and eliminate any variation of thickness. This tool can also be used to standardize the process of sanding glassy polymer samples to allow for greater consistency of results between research groups. The purpose of the research reported in this thesis is to consider the process of producing a dynamic mechanical analysis sample preparation housing.

CHAPTER TWO: LITERATURE REVIEW

One of the issues that was found in literature at the beginning of this project is the lack of information regarding DMA sample bar preparation. It is very challenging to compare results between research groups if each group attains the subject of analysis by different methods. In one article, McAninch et al. (2015) characterized five different epoxy networks using three different DMA techniques.¹⁴ The centerpiece of this article is the differences between glass transition temperature readings between the techniques and networks. Despite the entire article focusing on DMA sample geometries and their effect on Tg, the sample preparation of the samples is not described. The experimental section states, the epoxy-amine resin was degassed, poured into an aluminum mold, and cured. After the bars were cured, the samples were taken out of the mold and grinded down using a surface grinder to the appropriate specifications. Although mold release was used, members of the Wiggins research group found that using a hard mold makes removing the cured samples impossible without destroying the samples. There is no further explanation in the article of the method used to remove the samples.

An article by Yu-Hsuan Liao et al. (2004) investigates the mechanical effects of single-walled nanotubes in epoxy resin systems.¹⁵ In the experimental section, Liao mentions the polymer is placed into rectangular molds in which the polymer is to be cured. After a post-cure heating step, the bars are “polished and prepared for a single cantilever DMA analysis”. No further explanation of what “polished” entails or what the dimensions of the bars were for DMA testing. These inconsistencies in literature could be dealt with in future articles by using a standard preparation protocol and a specified tool that produces

consistent results. It would also be a benefit if the standard protocol produced consistent results quickly.

CHAPTER THREE: DESIGN PROCESS

The design objective for this project was to create a device and/or process to form rectangular bars of fully cured thermoset polymer into precise dimensions faster than hand sanding. The first concept that was proposed was a ‘clamp’ design. This design, pictured in Figure 4, is a DMA bar sanding method in which two mechanical arms hold the sample in place while two sanders on either side come together to sand the sides of the bar. The arms would be programmed to reorient after a set time so the sanders could sand the top

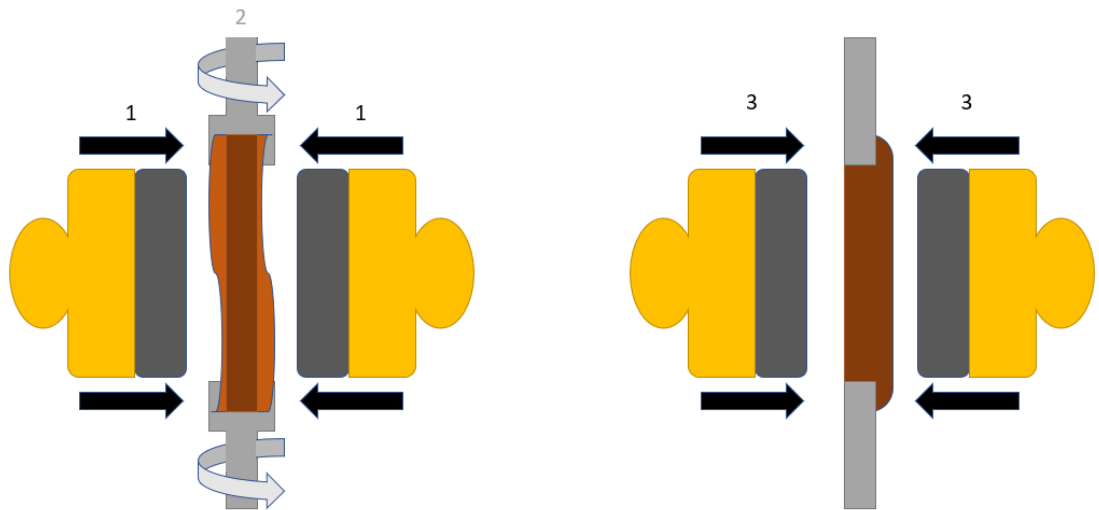


Figure 4, Clamp sanding design front view

and bottom of the bar. After deliberation, this concept was not pursued due to the sheer number of possible points of failure. There was a high possibility that the device would not sand correctly. The design team anticipated difficult troubleshooting from too many moving parts. Reorienting the mechanical arms on its own would be a difficult task, and an accurate proof of concept for the precision of the tool would not be possible until most of the instrument was built. Additionally, the sanders would be too large compared to the bars for such a device to be possible.

The second concept considered was a ‘carwash’ design. This design, pictured in Figure 5, shares features of the mechanical arm design with an important distinction, there

is no automation. In this design a housing with two sanders facing each other on an adjustable mount would be used. The bars would be placed into the mouth of the device and pushed through to the other side while the sanders shaved down the

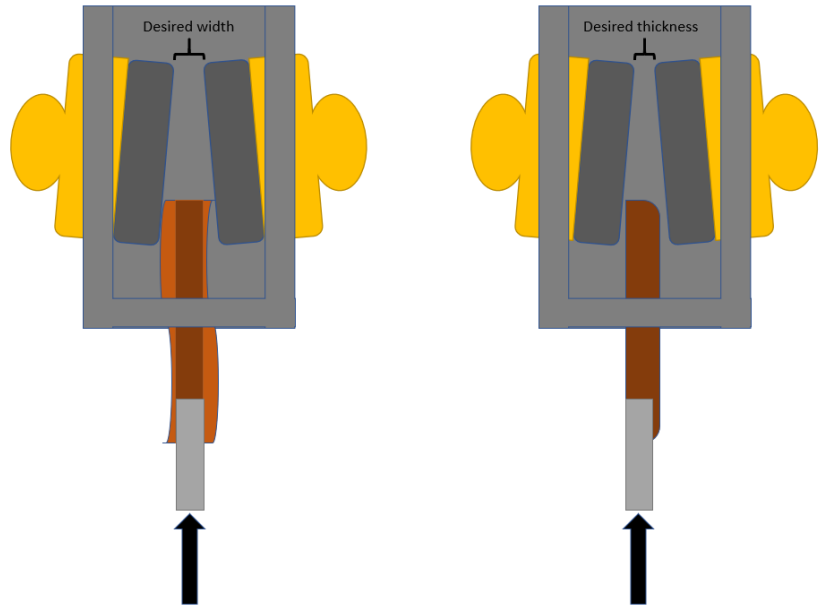


Figure 5, Carwash design top view cross-section

material to the specifications determined by the gap size between the sanders. A cover would be used to prevent the bars from ejecting from the apparatus. Although the mechanism is more controlled than the previous concept, the process seemed to be missing the core problem with the current DMA bars which is the presence of too much flash on the top of the bars. This flash can fuse multiple bars together, Figure 6. However, the other dimensions defined by the mold are correct. Sanding the sides and bottom of the bars would be unnecessary and ineffective at solving the issue.



Figure 6, Post-cured DMA bars

With this realization, the next concept tried to tackle the source of the issue. The ‘improved mold’ design sought to prevent the flash from occurring during curing. This concept proposed a new mold with a cover or simply a cover to the existing mold. This concept seemed to be the simplest solution to the problem; however, several major concerns prevented the group from continuing in this direction. The choice in material for the mold would be an issue. Silicone would be preferred due to its non-stick nature when in contact with polymer that is undergoing cure as well as its flexible nature allowing the mold to be bent, releasing the cured samples. The closed mold may prove to be an explosive hazard when curing exothermic systems especially if any volatile gases are trapped in the mold. There would also not be a way of minimizing the effect of polymer shrinkage. Additional sanding would still be needed if shrinkage or warpage occurred, rendering the mold ineffective.

The final design considered was named the ‘housing’ design. This design called for an aluminum housing with divots to hold DMA bars in place so an electric sander could sand off the excess flash on the top of the bars quickly with less variation in thickness. There were many considerations that were made before the embodiment of this design. A preliminary idea was to automate the process by using an electrical lift that would move the housing up to the sander for a set time limit for sanding. This mechanism is ideal but was too ambitious for the timeframe in which the project was to be completed. The second design feature considered stemmed from the possibility of difficult removal of the DMA bars from the housing after sanding. The prospective solution to this problem was to implement pushpins that sit in the cavity of the mold and can be pushed from the bottom

of the housing to release the bars. This feature was kept as a workable solution should the proposed issue manifest itself in initial testing.

The first prototype of the housing concept was initially designed in SolidWorks 2017, pictured in Figure 7. The prototype features a quarter circle block of radius 3 inches and height of 1 inch. The cavities are of length 60 mm, width 5 mm, and depth 0.9 mm. These dimensions were chosen because the dimensions of the post sanded bars were 60 mm by 5 mm, by 1.0 mm. The depth was decreased in order to prevent any possible scraping of the housing near the end of sanding the bar. The cavities were radially spaced because it was thought that the random orbit sander would produce smoother bars if the width of the bars were aligned with the movement of the sander. Inside corners are not

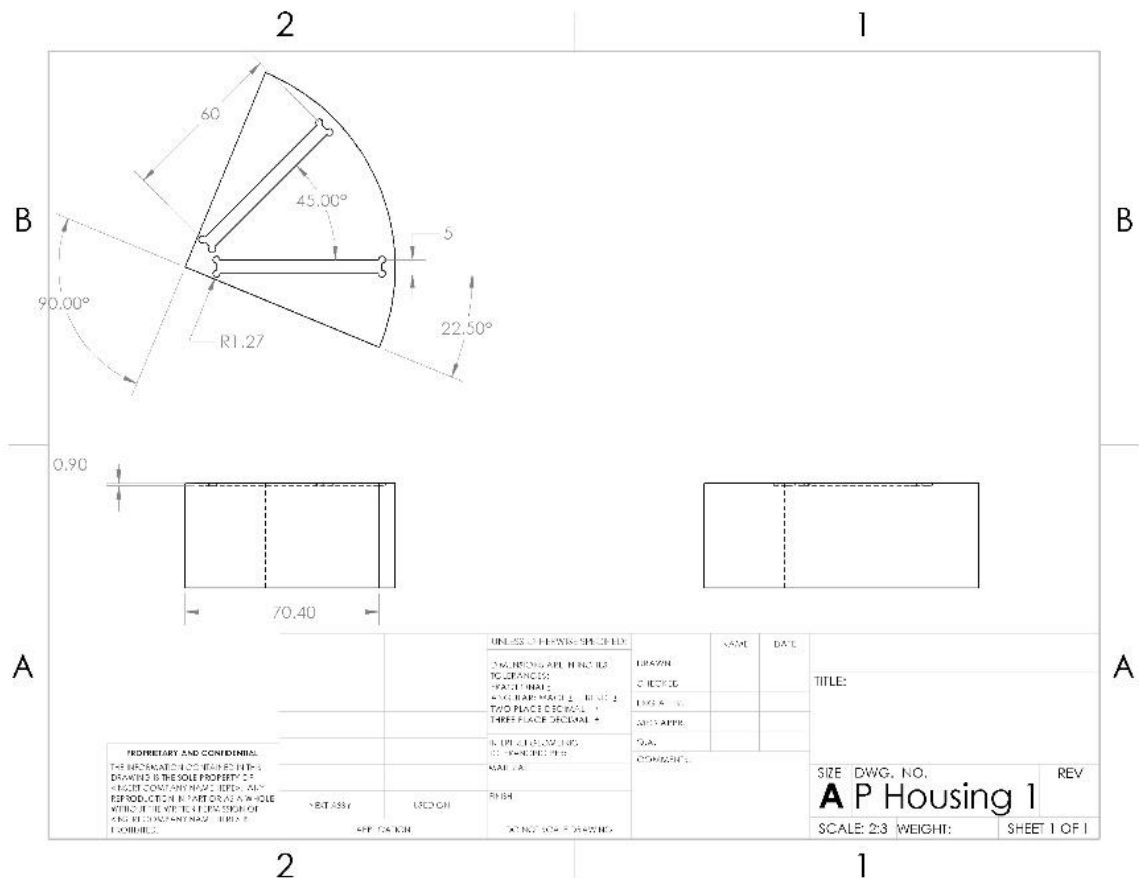


Figure 7, Prototype 1 drawing

possible to tool due to the geometry of taps that are used for tooling. To remedy this, 1.25 mm radius circular divots in the corners were added to the design. The part was tooled using Hardinge Bridgeport CNC machine using Master CAM and SolidWorks software. The tooling time was approximately 30 minutes. To reduce tooling time, the quarter circle block was changed to a simple rectangular block.

Once tooled, pictured in Figure 8, the part was tested using several tetraglycidyl methylenedianiline (TGDDM)/3,3-diamino diphenyl sulfone (3,3-DDS) epoxy-amine DMA bars to verify that the concept works. The

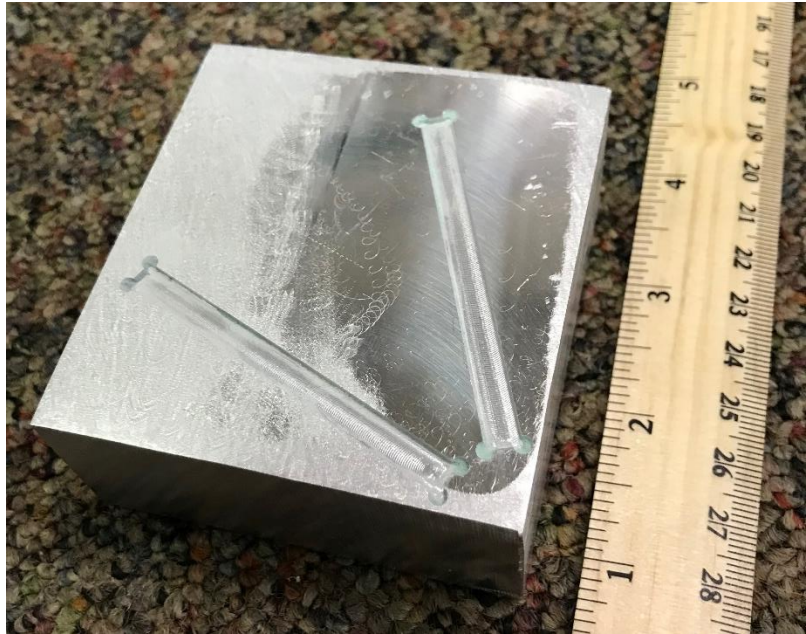


Figure 8, First prototype tooled

first prototype was bulky, because the block of aluminum was much thicker than was necessary. During these preliminary tests, one of the first concerns was that the bars were a few millimeters longer than the cavity. This was an issue in measuring the length that needed to be fixed in future prototypes. The bars were broken to a size that would fit into the cavity as a temporary solution to resume testing. A clamp was used to secure the housing in a fume hood. Once fitted, a DeWALT™ 2.3 Amp ¼ Sheet Palm Grip Sander with 80 grit mesh sandpaper was connected to a voltage regulator set to 84 volts. The first experiment focused on taking measurements of three different points of the bar using a

caliper, timing the sanding, and remeasuring the same points in order to calculate the rate of sanding. Unfortunately, every time the sander was applied the bars wiggled out of the housing after approximately 20 seconds which skewed the rate of sanding data. The sanding was promising, but a new feature needed to be added in order to prevent the bars from spontaneously ejecting from the housing. One additional note was the propensity for the sander to sand the edges of the bars down to a fine edge on the ends of the bars rather than only down to the depth of the cavity.

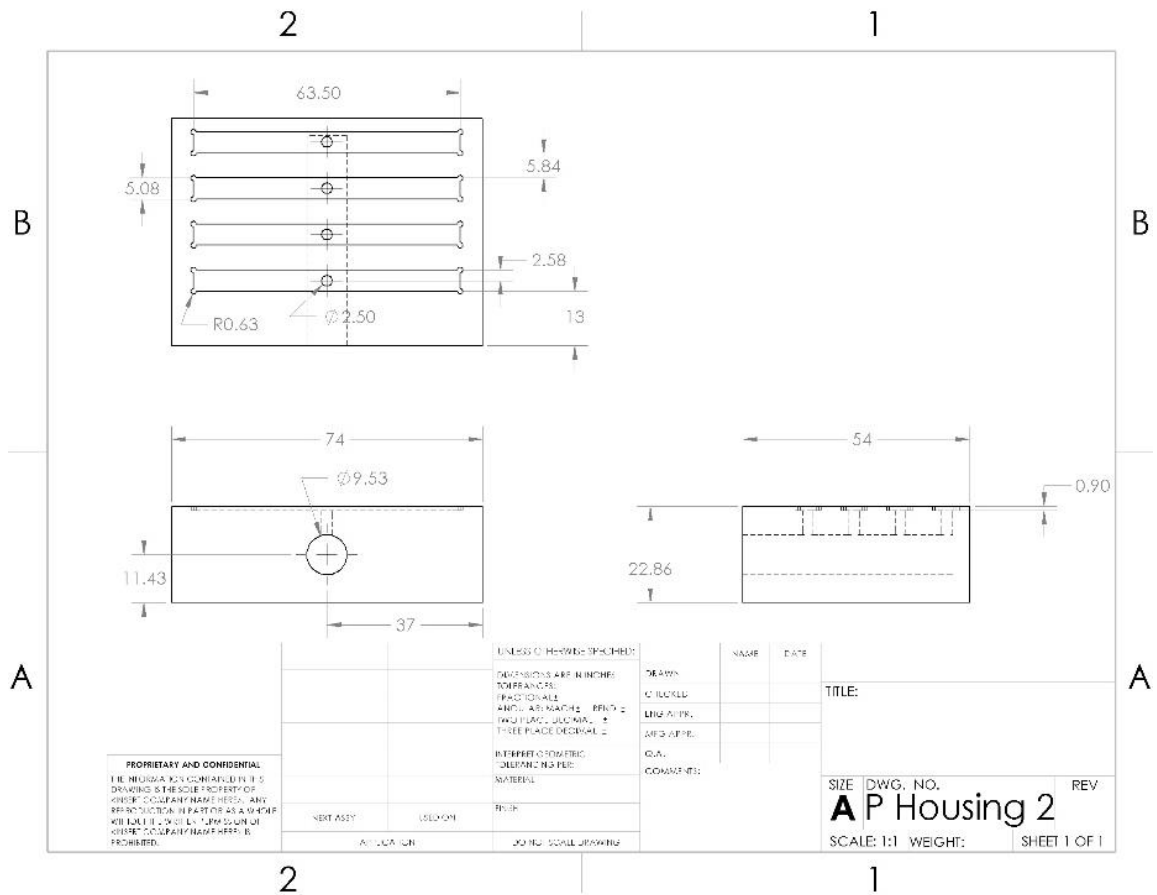


Figure 9, Prototype 2 drawing

The second prototype drawing pictured in Figure 9 and embodied in Figure 10 features a side-by-side cavity design mimicking the design of the DMA mold that is used for curing the samples. This allows more samples to be sanded, as well as samples that are

fused together during the curing process. The dimensions of the block are 74 mm x 54 mm x 23 mm. The dimensions of the four cavities are 63.5 mm x 5.1 mm x 0.9 mm. The length was extended by 3.5 mm to accommodate the bar length. The spacing between the cavities are 10.9 mm. This spacing distance was used because it was the distance between cavities of the curing mold. This prototype also features an internal vacuum system. Holes of diameter 2.5 mm were configured in the center of the cavities as ports for suction to hold down the samples during sanding. These ports are connected to a larger internal line of diameter 9.5 mm that is threaded to the dimensions for national pipe threading (NPT) to correspond with a 3/8 inch

laboratory nozzle from T&S Brass (B-0198-F03 Serrated Tip/Hose End Outlet, 0.25 GPM). This device can then be attached by a hose to a vacuum. To avoid dust from entering the vacuum, a cotton ball

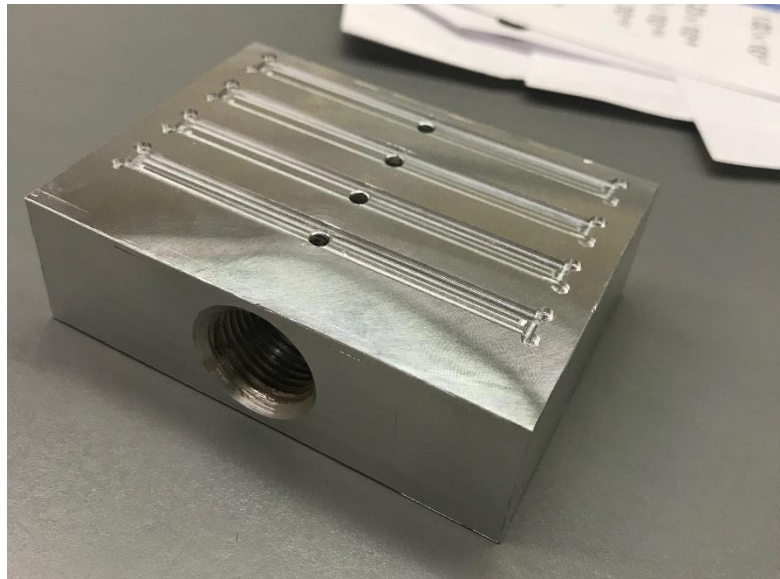


Figure 10, 2nd prototype tooled

was placed inside of the nozzle as a particulate filter.

The first set of experiments were called the various vacuum test. In the various vacuum test, two vacuums of different capabilities were used to test the necessary vacuum pressure needed for this application. The two vacuums used were a Fischer Scientific Maxima C Plus Model M12C (medium vacuum) and a traditional water aspirator pump (low vacuum). Measurements of the DMA bar thickness were taken at three different points

using calipers. Two points were taken approximately half an inch from each end of the bar. The third point was taken in the middle of the bar. Of the four cavities available, two were filled with pre-used bars that were thinner than 1 mm to cover the vacuum holes to hold the vacuum. The last two cavities were filled using two DMA bars of high thickness that were fused together. The palm sander was used at 84 volts and 120 volts to see if sanding power would influence the bar slippage. It was assumed that both sanders would yield similar results regarding slippage. The cotton ball placed in the nozzle was replaced after each sanding treatment to avoid saturation and skewed results. The sanding was done for 2 minutes. The thicknesses of the bars were then measured and recorded.

The second series of experiments was deemed the movement study. These tests studied the differences between stationary sanding and massaging the sander in circles over the entire housing for both the palm sander mentioned earlier and the DeWALT™ 3 Amp 5 in. Random Orbital Hook and Loop Sander with 80 grit sandpaper. For this study, the medium vacuum was used for all the runs, and the voltage remained constant at 84 volts. Calipers were used to measure the initial thickness at three points. Measurements were taken every 3 minutes to obtain the rate of sanding and to find out if the bars become more uniform in thickness over time. Two to three bars were tested with each sander. The tests were complete after 9 minutes for fused bars of two or 15 minutes for fused bars of three.

After several tests, the problems with the second housing became apparent. The largest problem using the sander was the placement of the vacuum ports on the housing. The bars managed to still wiggle out of the housing occasionally, lengthening the amount of time it took to sand the bars. Also, the bars exhibited the propensity to sand to a fine edge on the ends of the bars, as seen in Figure 12. This can be due minor warpage of the

polymer bars that cause the ends to flair upward toward the ends of the bars. Another large issue was of cavity spacing. The spacing used for the second prototype were direct measurements taken from the silicone mold. The spacing was too precise which prevented some bars, especially fused bars, from fitting into the cavities correctly. Due to the

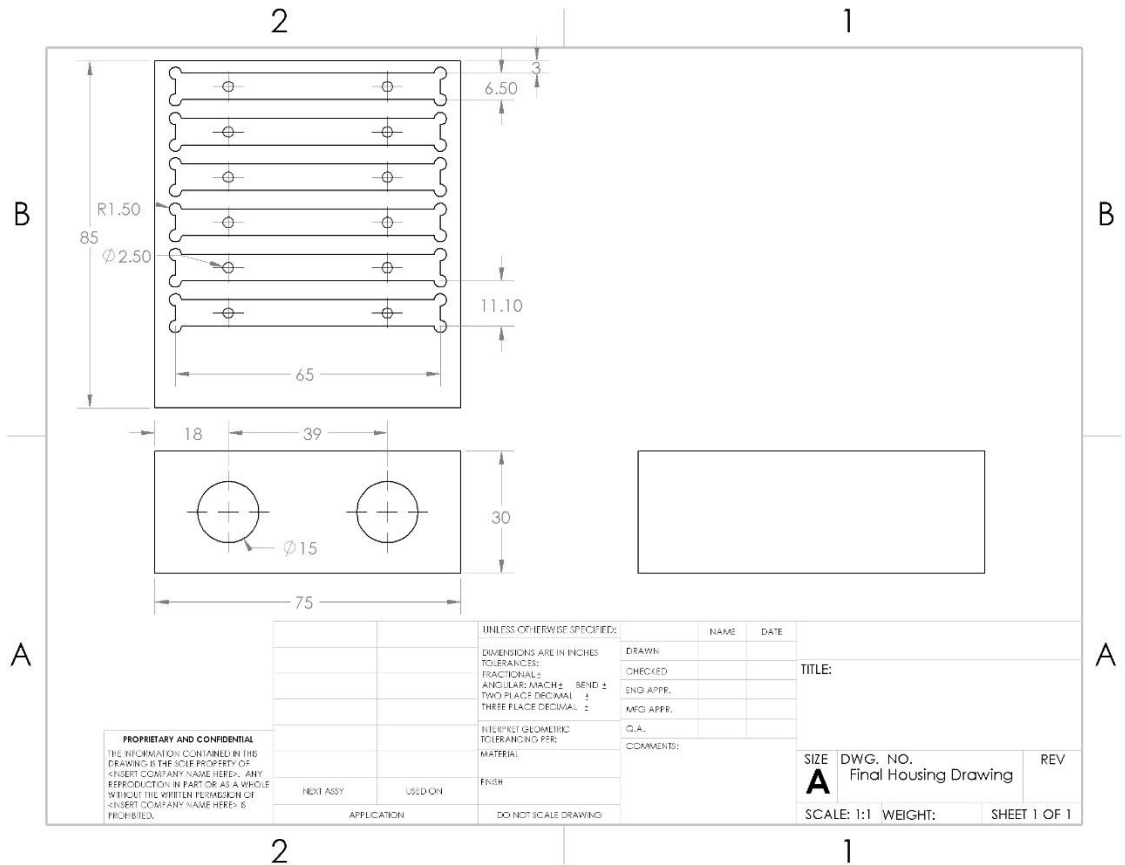


Figure 11, Final prototype drawing

bulkiness of the first housing, the second housing was made to be merely 23 mm thick. This size was much easier to handle. However, the nozzle used for the vacuum system was wider than the thickness of the housing. This prevented the housing from lying flat on a surface. This also made sanding more difficult because it required actively avoiding sanding the side of the nozzle.

The third and final iteration of the housing is pictured in Figure 11. This housing is of dimensions 75 mm x 85 mm x 30 mm with six cavities of 65 mm x 6.5 mm x 1 mm spaced 11.1 mm apart. A major feature change is the implementation of two independent vacuum ports of 15 mm diameter with vacuum holes of 2.5 mm

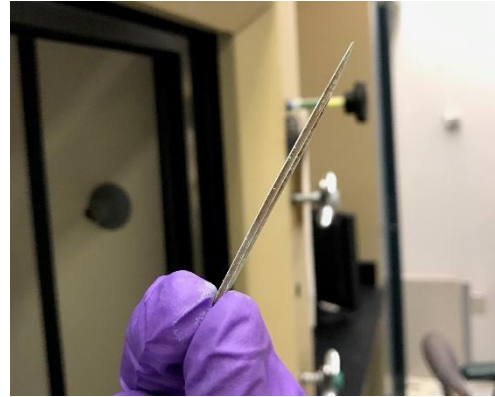


Figure 12, Sharpened DMA bar

diameter 13 mm from the ends of the bars to prevent slippage or over sanded edges. Other features include larger cavities and improved spacing based on fused bars out of the mold rather than the mold itself. Two extra cavities were added in order to sand more bars at the same time. The housing was thicker to account for the shape of the nozzles, allowing the housing to sit flat on surfaces. The depth of the cavities was increased from 0.9 mm to 1.0 mm to lessen the likelihood for the bar to be over-sanded from natural warpage.

CHAPTER FOUR: RESULTS AND DISCUSSION

The vacuum study test data is shown in Table 1. Unfortunately, accurate aspirator pump data could not be obtained due to the propensity of the samples to fall out of the mold. This frequent occurrence allowed the design group to rule out the aspirator pump as a viable vacuum option for our system. The medium vacuum fared much better; however, even while under vacuum the bars did not sit flat in the mold. The two connected samples that were used for testing did wiggle out when the sander was not placed directly on top of the bars with adequate force. The pre-sanded DMA bars used as place holders to ensure a strong vacuum also fell out multiple times at 120 volts and 84 volts. Both bar 1 and 2 showed signs of over-sanding on the right side. Solutions were found to problems that plagued the vacuum study, and they were implemented into the final prototype. These improvements include the new vacuum chuck placements, larger cavity sizes, improved cavity spacing based on measurements of fused bars rather than the silicone mold.

| Vacuum Study | | | | | | | |
|---------------------|--------------------|--------|-------|-------|--------|-------|--|
| Palm Grip | bar 1 | | | bar 2 | | | |
| | bar thickness (mm) | | | | | | |
| time (min) | left | middle | right | left | middle | right | |
| 0 | 2.54 | 2.35 | 2.08 | 2.42 | 2.23 | 1.99 | |
| 2 | 2.22 | 2.26 | 1.55 | 2.06 | 2.11 | 1.48 | |

Table 1, Vacuum study results

| Stationary vs Movement | | | | | | | |
|-------------------------------|--------------------|----------|----------|----------|----------|----------|------------|
| Palm Grip (Stationary) | | | | | | | |
| | bar 1 | | | bar 2 | | | |
| | bar thickness (mm) | | | | | | |
| time (min) | left | middle | right | left | middle | right | |
| 0 | 1.7 | 2.32 | 2.01 | 1.97 | 2.52 | 2.55 | |
| 3 | 1.74 | 2.33 | 2.02 | 1.98 | 2.38 | 2.26 | |
| 6 | 1.63 | 2.28 | 1.92 | 1.96 | 2.42 | 2.36 | |
| 9 | 1.37 | 2.3 | 1.96 | 1.93 | 2.33 | 2.27 | |
| avg (mm/min) | 0.036667 | 0.002222 | 0.005556 | 0.004444 | 0.021111 | 0.031111 | 0.01685185 |
| Palm Grip (Massage) | | | | | | | |
| | bar 1 | | | bar 2 | | | |
| | bar thickness (mm) | | | | | | |
| time (min) | left | middle | right | left | middle | right | |
| 0 | 1.37 | 2.3 | 1.96 | 1.93 | 2.33 | 2.23 | |
| 3 | 1.23 | 2.24 | 1.71 | 1.84 | 2.27 | 2.09 | |
| 6 | 1.13 | 2.22 | 1.72 | 1.64 | 2.21 | 1.96 | |
| 9 | 1.11 | 2.17 | 1.46 | 1.75 | 2.14 | 1.76 | |
| avg (mm/min) | 0.028889 | 0.014444 | 0.055556 | 0.02 | 0.021111 | 0.052222 | 0.03203704 |

Table 2, Palm sander movement study results

The palm grip movement study data is shown in Table 2. The inspiration for this study came from an anomalous occurrence while doing preliminary sanding studies. While sanding, one student held the palm sander completely still. The sanded bar exhibited odd wave-like topography pictured in Figure 13. This was troublesome because if it is not possible to attain an even thickness using a fixed sander, the prospects of automating the process or making a mechanical rig to simplify the process for the user become implausible. Luckily, the wave-like topography was a fluke and did not happen again during the movement studies. The random orbit movement data is in Table 3. The random orbit sander sanded 0.135 mm /min while stationary and 0.0759 mm /min

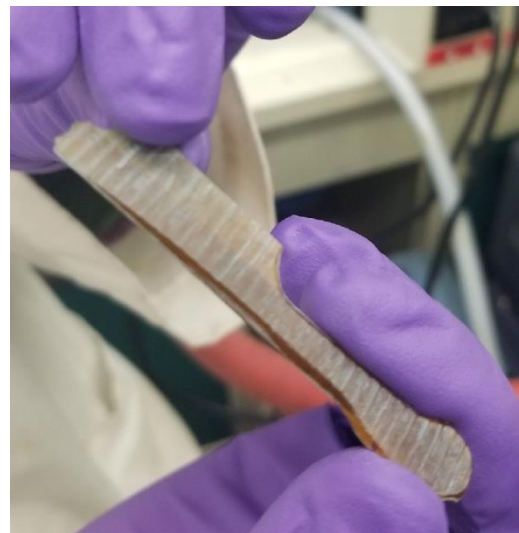


Figure 13, Wavy sample anomaly

while in-motion whereas the palm sander sanded 0.0169 mm /min while stationary and 0.0320 mm/min while in-motion. The random orbit sander performed nearly twice as well as the palm sander. As a result, the random orbit sander was used for the rest of the optimization studies. The circular motion experiments for both sanders exacerbated the problem of samples ejecting from the housing. The efficiency of the novel sanding technique was hampered by the time used to place the samples back into the housing after sample ejection. During the movement studies, avoiding the metal nozzle proved difficult. The metal nozzle also tilted the entire housing which may have caused the DMA bars to be sanded unevenly. As a result, the housing thickness was increased in the final iteration of the housing. Due to time constraints, the final iteration of the housing was not able to be tested.

| Orbital (Stationary) | | | | | | | | | | |
|-----------------------------|--------------------|----------|-------|----------|----------|----------|-------|----------|----------|----------|
| | bar 1 | | | bar 2 | | | | | | |
| | bar thickness (mm) | | | | | | | | | |
| time (min) | left | middle | right | left | middle | right | | | | |
| 0 | 1.97 | 2.17 | 1.54 | 2.1 | 2.16 | 1.47 | | | | |
| 3 | 1.09 | 1.21 | 1.17 | 1.57 | 1.94 | 1.45 | | | | |
| 6 | 1.08 | 1.2 | 1.15 | 0.82 | 1.15 | 1.15 | | | | |
| avg (mm/min) | 0.148333 | 0.161667 | 0.065 | 0.213333 | 0.168333 | 0.053333 | 0.135 | | | |
| Orbital (Massage) | | | | | | | | | | |
| | bar 1 | | | bar 2 | | | bar 3 | | | |
| | bar thickness (mm) | | | | | | | | | |
| time (min) | left | middle | right | left | middle | right | left | middle | right | |
| 0 | 2.32 | 2.53 | 2.68 | | | | 2.56 | 2.46 | 2.72 | |
| 3 | 2.17 | 2.23 | 2.25 | | | | 2.23 | 2.42 | 2.42 | |
| 6 | 1.98 | 2.14 | 1.87 | | | | 2.45 | 2.31 | 2.27 | |
| 9 | 1.33 | 1.67 | 1.72 | 1.49 | 2.09 | 2.13 | 1.65 | 2.03 | 2.15 | |
| 12 | 1.31 | 1.71 | 1.48 | 1.07 | 2.11 | 2.11 | 1.4 | 1.98 | 1.99 | |
| 15 | | | | 1.05 | 1.86 | 1.5 | 1.48 | 1.81 | 1.24 | |
| avg (mm/min) | 0.084167 | 0.068333 | 0.1 | 0.073333 | 0.038333 | 0.105 | 0.072 | 0.043333 | 0.098667 | 0.075907 |

Table 3, Random orbit sander movement study results

In conclusion, dynamic mechanical analysis is a sensitive instrumental analysis technique used for the characterization of several critical polymer properties. Being able to

better compare results and have a standard, simplified, and faster method for making DMA bars for testing is necessary. Currently, there is not a standard method of DMA sample preparation. Some research groups polish their samples and other groups have no mention of how the sample bars were made. The design solution this project has found is a sample housing that fixes the bars in place so that a palm sander can effectively and evenly remove excess polymer left on the samples. This can possibly eliminate the long turnover of hand-sanding samples made using a silicone mold. In the future, the team will run experiments to obtain the sanding rate of the final prototype. These results will be compared to hand-sanding rates. The variation of thickness of machine sanded bars will be compared to hand-sanded bars. Also, DMA will be conducted on samples obtained via machine sanding and will be compared to hand-sanded bars to find the average variation of glass-transition temperature.

REFERENCES

- (1) MarketsandMarkets. Commodity Plastics Market Worth 493.74 Billion USD by 2022 <https://www.prnewswire.com/news-releases/commodity-plastics-market-worth-49374-billion-usd-by-2022-670516893.html> (accessed Mar 10, 2019).
- (2) Inc, R. Viscosity of Newtonian and Non-Newtonian Fluids <http://www.rheosense.com/applications/viscosity/newtonian-non-newtonian> (accessed Mar 10, 2019).
- (3) Flow Properties of Polymers <http://polymerdatabase.com/polymer%20physics/Viscosity2.html> (accessed Apr 9, 2019).
- (4) Viscoelastic Behavior - an overview | ScienceDirect Topics <https://www.sciencedirect.com/topics/materials-science/viscoelastic-behavior> (accessed Mar 26, 2019).
- (5) Smithers, R. First Sustainable Lego Pieces to Go on Sale. *The Guardian*. March 2, 2018.
- (6) Challenger Explosion | ThinkReliability, Case Studies. *ThinkReliability*.
- (7) Mettler-Toledo International Inc. Investigation of The Bouncing Behavior of Two Rubber Balls https://www.mt.com/hk/en/home/supportive_content/matchar_apps/MatChar_UC404.html (accessed Mar 11, 2019).
- (8) Differential Scanning Calorimeter (DSC) TA Instruments Q100 – Wisconsin Centers for Nanoscale Technology – UW–Madison <https://wcnt.wisc.edu/soft-materials-characterization-lab/differential-scanning-calorimetry/ta-instruments-q100-differential-scanning-calorimeter/> (accessed Mar 27, 2019).
- (9) James, J. Chapter 7 - Thermomechanical Analysis and Its Applications. In *Thermal and Rheological Measurement Techniques for Nanomaterials Characterization*; Thomas, S., Thomas, R., Zachariah, A. K., Mishra, R. K., Eds.; Micro and Nano Technologies; Elsevier, 2017; pp 159–171. <https://doi.org/10.1016/B978-0-323-46139-9.00007-4>.
- (10) A Closer Look: Techniques for Obtaining Glass Transition Temperature of Polymeric Materials <http://www.intertek.com/blog/2013-04-15-glass-transition-temperature/> (accessed Mar 12, 2019).
- (11) Dynamic Mechanical Analysis (DMA) A Beginner’s Guide. 23.
- (12) Menard, K. P.; Menard, N. R. Dynamic Mechanical Analysis in the Analysis of Polymers and Rubbers. In *Encyclopedia of Polymer Science and Technology*; American Cancer Society, 2015; pp 1–33. <https://doi.org/10.1002/0471440264.pst102.pub2>.
- (13) Varley, R. J.; Dao, B.; Tucker, S.; Christensen, S.; Wiggins, J.; Dingemans, T.; Vogel, W.; Marchetti, M.; Madzarevic, Z. Effect of Aromatic Substitution on the Kinetics and Properties of Epoxy Cured Tri-Phenylether Amines. *J. Appl. Polym. Sci.* **2019**, *136* (18), 47383. <https://doi.org/10.1002/app.47383>.
- (14) McAninch, I. M.; Palmese, G. R.; Lenhart, J. L.; Scala, J. J. L. DMA Testing of Epoxy Resins: The Importance of Dimensions. *Polym. Eng. Sci.* **2015**, *55* (12), 2761–2774. <https://doi.org/10.1002/pen.24167>.
- (15) Liao, Y.-H.; Marietta-Tondin, O.; Liang, Z.; Zhang, C.; Wang, B. Investigation of the Dispersion Process of SWNTs/SC-15 Epoxy Resin Nanocomposites. *Mater. Sci. Eng. A* **2004**, *385* (1), 175–181. <https://doi.org/10.1016/j.msea.2004.06.031>.