MECHANICAL PROPERTIES OF THICK WALL, COMPRESSION MOLDED PEEK AT ELEVATED TEMPERATURES

A Thesis

by

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MASTER OF SCIENCE

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ABSTRACT

The oil-and-gas industry uses PEEK (polyether ether ketone) because PEEK withstands high temperature and pressure. High temperature and pressure typically degrade and enhance—respectively—polymer mechanical properties from the room temperature values. It is important to build a test apparatus that measures PEEK's mechanical properties under these conditions. This work is the first step in creating such an apparatus by testing thick-wall compression-molded PEEK at elevated temperatures. At room temperature, yield strength was within experimental error between compression molded PEEK samples and injection molded samples at 0.1 s⁻¹ strain rate. More work is needed to compare PEEK's mechanical properties when using different process methods.

CONTRIBUTORS AND FUNDING SOURCES

Contributors

This work was supervised by a thesis committee consisting of Professor Creasy and Professor Muliana of the Department of Mechanical Engineering and Professor Sue of the Department of Materials Science.

The samples used in this work were prepared in part by Ruaa Al Mezrakchi of the Department of Mechanical Engineering.

All other work conducted for the thesis was completed by the student independently.

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1. INTRODUCTION

PEEK (polyether ether ketone) is an important thermoplastic used in oil and gas applications because PEEK's high stiffness, high strength, wear resistance, and chemical resistance are good at elevated temperature and pressure [1]. High temperature and high pressure applied to polymers often produce competing effects: high temperature typically decreases the yield strength while high pressure can increase the yield strength [2] [3]. Because these effects counter each other, PEEK should be under combined temperature and pressure when characterized.

Characterization—and most part production—at this time uses injection molded specimens or specimens machined from commercially made sheet formed under unknown processing conditions. Processing can give PEEK crystallinity anywhere between 0 to 40 percent [1]. Injection molding creates less than ideal properties if the mold temperature is too low. Annealing can partially correct the crystallinity, but annealing can adversely affect other properties [1]. Compression molding forms PEEK under different conditions (time, temperature, pressure) that influence mechanical properties [4]. Oil and gas applications use compression molding for ring seals because weld lines that can occur in injection molded rings create weak spots.

Determining accurate properties for PEEK under these conditions has not happened. It is important to create an apparatus that can perform tests under extreme conditions to give engineers better properties for design and material lifetime. This work takes the first step towards this goal by examining PEEK's mechanical properties in an elevated temperature environment while using processing conditions that closely resemble production methods for oil and gas applications.

2. LITERATURE REVIEW

Many factors affect PEEK's mechanical properties. Yuan et al. looked at molecular weight [5]. Ultimate tensile strength increases with increases in molecular weight. Yet tensile modulus decreased as the molecular weight increased and tensile yield strength decreased slightly with increased molecular weight.

Shrestha et al. studied fatigue effect on PEEK [6]. PEEK softens when exposed to uniaxial strain-controlled loading in tension and compression. Compared to metals, PEEK's fatigue response to load-controlled cycling and to strain-controlled cycling was interesting because load-controlled cycling generates smaller strain response than obtained with the same stress applied under strain-controlled cycling.

Several experiments investigated PEEK's flexure response with temperature. Adams et al. investigated flexural modulus from ambient temperature to -196 °C [7]. The flexure specimens were 200 by 13 by 3mm and bought from ICI, the company that invented PEEK. The stiffness increased as temperature decreased. Schwitalla et al. performed 3-point bending tests on samples stored dry or in Ringer solution at 37 °C for 1, 7, 28, and 84 days and then tested at room temperature. Extrusion, injection molding, and pultrusion methods formed the specimens [8]. Experiments evaluated several PEEK compounds with added titanium oxide or carbon fiber. The compound properties stayed the same despite the immersion time.

Rae et al. studied 450G PEEK with compression tests from -85 to 200 °C and at strain rates from 10^{-4} to 3000 s⁻¹ [2]. The author used an extruded 19 mm-thick plate to

create specimens. Differential scanning calorimetry determined crystallinity was 41 percent. Heating or cooling the compression platens controlled the temperature. Rae et al. created cylindrical samples with a 1:1 aspect ratio. The author deformed the samples to large strains. Rae et al. performed hardness, fracture toughness, impact, and tensile tests. Over strain rates covering 10 orders of magnitude, the yield strength increased 30 percent with increasing strain rate. Elastic modulus changes little below glass transition temperature whatever temperature or strain rate are applied. Tests showed yield strength decreasing as temperature increased. This is like other semi-crystalline polymers.

Hamdan et al. studied compression properties in PEEK 150G purchased from ICI Plastics. Hamdan et al. created the specimens from 3.5 mm thick plate and tested at strain rates from 10^{-3} to 10^3 s⁻¹ and between 20 and 200 °C [9]. Below the glass transition temperature behavior was insensitive to strain rate. The yield strength decreased as temperature increased up to the glass transition temperature. Hamdan et al. found that more crystallinity formed above Tg temporarily increases PEEK's strength.

El-Qoubaa et al. investigated PEEK's compressive yield stress from -130 to 200 °C using strain rates 0.001, 0.1, and 5 s⁻¹ [10]. El-Qoubaa et al. bought the samples as 10 mm cylindrical rods from ENSINGER France and were 450G. Compressive yield stress was highly affected by low temperature and high strain rates.

There are several earlier studies in tribology on PEEK. Laux et al. examined contact temperature in PEEK with steel and sapphire counter faces [11]. Zalaznik et al. showed that processing temperature is an influential factor in PEEK mechanical properties [4]. Changing PEEK's processing temperature between 300, 350, and 400 °C

using compression molding allowed the researchers to mimic different processing conditions. The study examined hardness and other tribology related properties. As the processing temperature increased the hardness increased. With the increase in hardness, Zalaznik et al. saw a higher wear rate.

Rau et al. examined PEEK in compression through Taylor cylinder impact testing [12]. The PEEK samples saw ductile deformation at the impact location and Xray tomography indicated a potential reduction in crystallinity. In addition, impact produced internal cracks in PEEK as opposed to PTFE samples that formed cracks at the impact location.

These results show that earlier compression test results are insufficient to characterize PEEK components manufactured in the oil and gas industry. Instead, the earlier compression test results will serve as a baseline for comparing results from study.

3. APPARATUS

There is a need for a self-contained apparatus to perform tests that model downhole conditions. Shown in Figure 1, the prototype apparatus major components are a frame, a stepper motor, a drive train, a load cell, a compression fixture, a voltage data logger and other electronic devices.

Three steel rectangular plates with two rods that allow the center plate to slide up and down make up the frame. The top plate has two threaded holes that the sliding rods screw into. The top plate has holes for the two ball screws and the stepper motor to allow connection to the drive train system. The middle plate has two plastic sleeves that allow the plate to slide up and down with reduced friction along the rods. The ball screw nuts and load cell mount to the middle plate. The bottom plate has threaded holes to attach two clamps that secure the sliding rods to the bottom plate. One sliding rod assembly appears shown in Figure 2.



Figure 1. Apparatus within a convection oven



Figure 2. Sliding rod assembly

An OMHT34-485 stepper motor attached to the top plate drives the apparatus. The stepper motor moves 2.5 degrees per step. The stepper motor speed was 0.1 rev/s. From the torque curve shown in Figure 3, the speed used is not on the torque curve. However, the motor was able to generate enough torque to plastically deform the samples and allow the yield strength to be determined.

The stepper motor drives a 9-tooth gear and two 72 tooth gears chain assembly attached to the ball screws with couplings. This gear ratio is 8, which means the motor torque increased by 8. The chain tensor tightens the chain to ensure good contact between the gears and chain. The ball screws move the nut 5.16 mm/rev. This allows calculating the distance traveled by a single motor step and that allows the strain on each sample to be determined. Figure 4 shows the ball screw assembly.



Figure 3. Stepper motor torque curve. Reprinted from Omega Engineering [13]



Figure 4. Ball screw and ball nut

The apparatus used a load cell to measure the force applied to the specimens. The load cell used has a 10 kN capacity. Bolts and a connector plate mounted the load cell to the middle plate. The load cell pushed on the compression fixture, which applied displacement to the specimen as shown in Figure 5.

The manufacturer supplied a load cell calibration table, which appears as Table 1. The calibration was checked by applying a calibration resistor across the -out to +exc terminals. The load cell excitation voltage was 10 Volts. The calibration curve shows the relationship between force and output voltage.



Figure 5. The load cell assembly

| Test Load (kN) | Compression Signal (mV/V) |
|-------------------|------------------------------|
| 0 | 0.0000 |
| 2 | -0.4185 |
| 4 | -0.8371 |
| 6 | -1.2557 |
| 8 | -1.6742 |
| 10 | -2.0927 |
| 4 | -0.8370 |
| 0 | 3 x 10 ⁻⁵ |

Table 1. Load cell calibration data. Reprinted from Interface Force Measurement Solutions [14]

Steel parts bolted together make up the compression fixture. The fixture works through a plunger in a sleeve with a hemisphere head that a plate with a hemisphere cutout sits on to ensure compression is only in the axial direction. Figure 6 shows the compression fixture.

The data logger measures +/- 30 mV change in voltage. The data logger's sampling rate was 4 Hz, the fastest allowed by the data logger.

A 48V power supply attaches to the Driver ST10-Si that runs the stepper motor. This controller programs the stepper to take steps at a specific speed. Here, the step count and speed were identical for each experiment. A voltage display and strain gage amplifier supply real time feedback about the sample load. The display's primary purpose was for experiment set up. Appendix B shows a circuit diagram for the electronics. Figure 7 shows the electronic set up.



Figure 6. Compression fixture



Figure 7. Electronics for the apparatus

4. EXPERIMENTAL PROCEDURES

The crosshead speed used during sample compression testing was determined experimentally and theoretically. A dial indicator and a stopwatch determined the time taken for the crosshead to move 2.54 mm. Table 2 displays the results with a 0.0624 mm/sec average crosshead speed.

The crosshead speed was also determined theoretically by multiplying the motor speed by the gear ratio and the distance traveled per revolution by the nut using this equation:

$$\frac{0.1 \, rev}{s} \ge \frac{1}{8 \, gear \, ratio} \ge \frac{5.16 \, mm}{rev} = 0.0645 \, mm/\sec (1)$$

This is a 3.26 % error.

Repeating the same experiment with a sample inside the compression fixture at ambient temperature led to different results. The dial indicator showed the crosshead speed changed during the test compared to without a sample. Table 3 displays the results.

| Table 2. Crosshead speed test results without specime | 'n |
|---|----|
|---|----|

| Trial Number | Time (s) |
|--------------|-------------|
| 1 | 41.51 |
| 2 | 40.15 |
| 3 | 41.38 |
| 4 | 39.76 |
| Average | 40.70 |

Table 3. Crosshead speed results with specimen

| Time Interval (s) | Crosshead Speed (mm/s) |
|----------------------|---------------------------|
| 0-10 | 3.3 x 10 ⁻² |
| 10-20 | 3.9 x 10 ⁻² |
| 20-30 | 5.3 x 10 ⁻² |
| 30-40 | 5.1 x 10 ⁻² |
| 40-49 | 3.3 x 10 ⁻² |
| Overall | 4.2 x 10 ⁻² |

The same crosshead speed settings led to a speed that was 35% slower than the test without a sample. The crosshead speed varied throughout the test; speed was slower at the beginning and with faster crosshead speed in mid travel.

The source material for the PEEK compression samples was a compression molded bushing. A band saw, set perpendicular to the bushing height, cut the bushing into slices [15]. A numbering system starting from the top, basing the processing orientation on the bushing label, showed each slices position. Once the band saw turned the bushing into slices, samples were CNC machined from the slices. The sample preparation procedure involved milling both slice sides flat and milling a hole in the slice to reduce the sample height to 6 mm. Milling the material around the sample until only 1 mm remained at the bottom allowed the sample's removal by a Dremel tool. A label on each sample showed the slice it came from.

To ensure that the entire sample is at 50 °C during each elevated temperature test, an experiment determined how long it takes the sample to reach 50 °C. Once the chamber reached steady state at 50 °C, a thermocouple between 2 specimens 3 mm thick each was placed inside the oven as show in Figure 8.



Figure 8. Specimen temperature test setup

The thermocouple reached 50 °C after 11 minutes. Therefore, during each elevated temperature test the compression samples remained in the test fixture for 11 minutes to soak up heat before each experiment.

A drift test performed on the load cell determined temperature effects on the signal. The apparatus was placed in the convection oven and heated to 50 °C. Data was recorded for 50 minutes, and the results are displayed in Figure 9. The results show the temperature related drift to be insignificant compared to the signal. Nevertheless, before starting elevated temperature testing, the load cell sat in the test chamber for 40 minutes before testing began.

A universal Insight test machine performed compression tests at 0.1 s^{-1} strain rate. The sampling rate was 10 Hz. The test fixture was compression platens. A convection oven heated the test chamber for the 50 °C tests. Each sample received 3 N preload before each test. An Instron test machine performed compression tests. The strain rate was 0.011 s^{-1} to match the apparatus crosshead speed without a sample. Each sample had a 3 N before the test. For elevated temperature tests the samples were in the hot test chamber for 11 minutes before the test started.

To perform tests on the self-contained apparatus, a compression sample was loaded into the test fixture. The gear drive assembly was hand driven until the load cell voltage display had a nonzero reading. The operator reversed the assembly until the system showed zero-load point. The data logger program was started. After 30 seconds the power supply for the stepper motor received power and the test was performed. Figure 10 displays the program used to run each test.



Figure 9. Load cell drift test at 50 °C

| Applied Motion | stilo-si 💽 | Help Line | | Description | Clear |
|--------------------------------------|------------|-----------|----|------------------------|-------|
| Products | Drive ? | 1 | | CW 94000 steps, .1 rps | |
| | Download | 2 | + | [| |
| | Upload | 3 | 4 | Go to line 2 | |
| Motor Encoder | Execute | 4 | + | [| |
| | Save | 5 6 | + | | |
| Alarms | Open | 7 | ¥ | | |
| 20000 Share dans | Print | 8 | + | [| |
| 20000 Stepsnev | Quit | 9 | + | [| |
| Jog Parameters peed 4.000 rev/sec | UserUnits | 10 | ♦ | [| |
| -] | name inch | 11 | + | | |
| ccel 25 rev/s/s | steps/inch | 12 | + | | |
|] | 20000 | 13 | + | | |
| | | 14 | | | |
| Configure I/O | 100 | 15 | .↓ | | |
| COM port | | 16 | + | | |
| _ | | 17 | + | | |

Figure 10. Stepper motor program

The stepper moves 94,000 steps; these steps move the crosshead 3 mm downward. The average strain rate was 0.007 s^{-1} . After moving the crosshead, the program enters an infinite, inactive loop where it waits until power goes off. Following testing, sample dimensions were measured and compared to the initial dimensions.

5. RESULTS

Table 4 shows the initial and final dimensions from the Insight specimens. Table 5 shows each specimen's initial and final dimensions when run on the Instron. Table 6 displays the self-contained apparatus test specimen dimensions. These results show that plastic deformation occurred in all specimens. Figure 11, Figure 12, and Figure 13 show the stress-strain curves from the PEEK compression tests at ambient temperature. The stress-strain curves for the PEEK compression tests specimens at 50 °C appear in Figure 14, Figure 15, and Figure 16. Table 7 and Table 8 show the PEEK's elastic modulus and yield strength determined by different test machines.

| Specimen # | Initial diameter (mm) | Initial length (mm) | Final diameter (mm) | Final length (mm) | Slice # | Temperature |
|---------------|-----------------------------|---------------------------|---------------------------|-------------------------|---------|-------------|
| 1 | 5.97 | 6.00 | 8.82 | 2.96 | 4 | Ambient |
| 2 | 5.89 | 5.83 | 9.26 | 2.58 | 3 | Ambient |
| 3 | 5.83 | 5.77 | 9.60 | 2.50 | 17 | Ambient |
| 4 | 5.96 | 5.80 | 9.46 | 2.57 | 17 | 50 °C |
| 5 | 5.88 | 5.85 | 9.39 | 2.61 | 4 | 50 °C |
| 6 | 5.87 | 5.76 | 9.56 | 2.58 | 17 | 50 °C |

Table 4. Insight specimen results

| Specimen | Initial diameter | Initial length | Final diameter | Final length | | |
|----------|---------------------|-------------------|-------------------|--------------|---------|-------------|
| # | (mm) | (mm) | (mm) | (mm) | Slice # | Temperature |
| 1 | 5.99 | 6.33 | 6.28 | 5.57 | 12 | Ambient |
| 2 | 5.98 | 6.33 | 6.01 | 5.97 | 12 | Ambient |
| 3 | 5.98 | 6.33 | 6.27 | 5.72 | 12 | Ambient |
| 4 | 5.99 | 6.33 | 6.26 | 5.66 | 12 | Ambient |
| 5 | 5.94 | 6.28 | 6.26 | 5.62 | 12 | 50 °C |
| 6 | 5.98 | 6.23 | 6.34 | 5.53 | 12 | 50 °C |
| 7 | 5.95 | 6.24 | 6.24 | 5.65 | 12 | 50 °C |
| 8 | 6.00 | 6.48 | 6.33 | 5.60 | 12 | 50 °C |

Table 5. Instron specimen results

| Specimen # | Initial diameter (mm) | Initial length (mm) | Final diameter (mm) | Final length (mm) | Slice # | Temperature |
|---------------|-----------------------------|---------------------------|---------------------------|-------------------------|---------|-------------|
| 1 | 5.84 | 5.92 | 6.86 | 4.67 | 4 | Ambient |
| 2 | 5.82 | 5.77 | 6.68 | 4.60 | 6 | Ambient |
| 3 | 5.84 | 5.56 | 6.78 | 4.29 | 6 | Ambient |
| 4 | 5.87 | 5.69 | 6.96 | 4.34 | 17 | 50 °C |
| 5 | 5.87 | 5.87 | 6.81 | 4.52 | 6 | 50 °C |
| 6 | 5.87 | 5.94 | 6.93 | 4.57 | 3 | 50 °C |
| 7 | 6.00 | 5.94 | | | 12 | Ambient |

Table 6. Self-contained apparatus specimen results



Figure 11. Insight stress-strain curve at ambient temperature



Figure 12. Instron stress-strain curve at ambient temperature



Figure 13. Self-contained apparatus stress-strain curve at ambient temperature



Figure 14. Insight stress-strain curve at 50 °C



Figure 15. Instron stress-strain curve at 50 °C



Figure 16. Self-contained apparatus stress-strain curve at 50 °C

Table 7. PEEK elastic modulus (MPa) at ambient and 50 °C temperatures.

| Temperature | Insight | Instron | Apparatus |
|-------------|---------|---------|-----------|
| Ambient | 2.64 | 1.93 | 1.21 |
| 50 °C | 2.18 | 1.88 | 1.26 |

Table 8. PEEK yield strength (MPa) at ambient and 50 °C temperatures.

| Temperature | Insight | Instron | Apparatus |
|-------------|---------|---------|-----------|
| Ambient | 153 | 140 | 142 |
| 50 °C | 132 | 122 | 125 |

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6. DISCUSSION

At this stage several improvements must be made to the self-contained device. A rigid drive train should replace the chain. The chain could jump a tooth while the motor was running. A completely rigid drive assembly would help ensure that all the force generated by the stepper motor is going into the sample with as little loss or rate variation.

An upgrade to the stepper motor must run at temperatures above 50 °C. The new stepper motor must run at 204 °C—closer to the conditions PEEK encounters in a downhole environment—to give accurate mechanical properties that PEEK has at that temperature. The new stepper motor will need to reach speed faster to supply a uniform strain rate during the experiment. The current stepper motor produced speed 37 percent under the target speed when the displacement started and ended.

Another improvement would be measuring strain directly. Currently the average motor speed determines the strain using a fixed speed throughout the experiment. Results show that is inaccurate as the speed varies with slow crosshead speed at the test's beginning and end.

A smaller apparatus is needed. The current size is 203 mm by 300 mm by 559 mm. Because future high-pressure testing will occur, this size must fit inside a pressure vessel where every cubic millimeter is crucial.

Future tests need a data logger with an increased sampling rate. The current data logger collects samples at 4 Hz. The universal testing machines collect data at 10 Hz.

The low sampling rate in the self-contained apparatus forced the experiments to be slow—slow enough to supply enough data for analysis. The current set up allows for x data points per test. Upgrading to a 10 Hz resolution would allow the experiment to run closer to the ASTM standard for compression tests.

In the future, components with higher temperature ratings must be used. The current ball screws have a 149 °C maximum temperature rating. The stepper motor has a 50 °C maximum operating temperature, and the load cell has a 90 °C maximum operating temperature. These all will need to increase to performed tests closer to downhole conditions.

In the literature El-Qoubaa et al. found that injected molded PEEK at a 0.1 s⁻¹ strain rate at ambient temperature to yield at 147 MPa [10]. This is within 5 percent of the value measured here for a compression molded sample at the same temperature and strain rate. The difference in process method does not appear to affect the PEEK's yield strength at ambient temperature. More testing is needed to determine if there is a difference in behavior at other temperatures and strain rates.

Compression molded PEEK sample yield strength decreased as expected with increasing temperature. The PEEK samples on the universal testing machines decreased in elastic modulus with as temperature increased. The self-contained apparatus samples had a slight increase in elastic modulus with increasing temperature. Each test machine produced a different elastic modulus, which either comes from calibration problems or rate dependent stiffness—different strain rates were used on each machine. Rate dependence might have caused the difference between the Instron and Insight testsboth are calibrated, professional machines. The Instron and self-contained apparatus have similar strain rates, but the results show a much lower elastic modulus comes from the self-contained apparatus. This might be caused by the method used to calculate strain, and, as described previously, a more direct measurement method is needed for future testing.

7. CONCLUSION

A first-generation apparatus that might perform compression tests at high temperature and pressure was built with PEEK samples tested at ambient and elevated temperatures. Changes to the data acquisition system, the drive train system, and strain measurement are improvements that can create a more reliable apparatus for future testing. Injected molded PEEK and compression molded PEEK samples were found to have the same compression yield strength at ambient temperature. More testing is needed to determine how mechanical properties change over a wider temperature and strain rate ranges.

REFERENCES

[1] Kemmish, D., 2010, Update on the technology and applications of polyaryletherketones, iSmithers, Shawbury

[2] Rae, P. J., Brown, E. N., and Orler, E. B., 2007, "The mechanical properties of poly(ether-ether-ketone) (PEEK) with emphasis on the large compressive strain response," Polymer, **48**, pp. 598-615.

[3] Mears, D. R., Pae, K. D., and Sauer, J. A., 1969, "Effects of Hydrostatic Pressure on the Mechanical Behavior of Polyethylene and Polypropylene," Journal of Applied Physics, **40**, pp. 4229-4237.

[4] Zalaznik, M., Kalin, M., and Novak, S., 2016, "Influence of the processing temperature on the tribological and mechanical properties of poly-ether-ether-ketone (PEEK) polymer," Tribology International, 94, pp. 92-97.

[5] Yuan, M., Galloway, J. A., Hoffman, R. J., and Bhatt, S., 2010, "Influence of Molecular Weight on Rheological, Thermal, and Mechanical Properties of PEEK,"
2011, Polym. Eng. Sci., 51, pp. 94-102.

[6] Shrestha, R., Simsiriwong, J., Shamsaei, N., and Moser, R. D., 2016, "Cyclic deformation and fatigue behavior of polyether ether ketone (PEEK)," International Journal of Fatigue, **82**, pp. 411-427.

[7] Adams, R. D., and Gaitonde, J. M., 1993, "Low-temperature flexural dynamic measurements on PEEK, HTA and some of their carbon fibre composites," Composites Science and Technology, **47**, pp. 271-287.

[8] Scwitalla, A. D., Spintig, T., Kallage, I., and Muller, W., 2015, "Flexural behavior of PEEK materials for dental application," Dental Materials, **31**, pp. 1377-1384.

[9] Hamdan, S., and Swallowe, G. M., 1996, "The strain-rate and temperature dependence of the mechanical properties of polyetherketone and polyetheretherketone," Journal of Materials Science, 31, pp. 1415-1423.

[10] El-Qoubaa, Z., and Othman, R., 2016, "Strain rate sensitivity of

polyetheretherketone's compressive yield stress at low and high temperatures,"

Mechanics of Materials, 95, pp. 15-27.

[11] Laux, K. A., Jean-Fulcrand, A., Sue, H. J., Bremner, T., and Wong, J. S. S., 2016,"The influence of surface properties on sliding contact temperature and friction for polyetheretherketone (PEEK)," Polymer, 103, pp. 397-404.

[12] Rau, C., Parry S., Garcea, S. C., Bourne, N. K., McDonald, S. A., Eastwood, D. S.,

Brown, E. N., and Withers, P. J., 2017, "On compression and damage evolution in PTFE and PEEK," *AIP Conference Proceedings*, **1793**, pp. 140006-1-4.

[13] 2018, "Performance Stepper Drives with Advanced Features and Control Options." from https://www.omega.com/auto/pdf/ST_SERIES.pdf

[14] 2017, "Load Cell Calibration Certification," Interface Force Measurement Solutions.

[15] Al Mezrakchi, R., 2018, Graduate Student at Texas A&M University, U.S.A., private communication.

APPENDIX A

User Instructions

- 1. Measure the diameter and length of the specimen
- 2. Plug in power supply for the strain gage amplifier and set to 18V.
- 3. Plug in voltage display.
- 4. Load and center a sample in the test fixture.
- 5. Hand turn the ball screws by one of the couplings until a there is a reading on the voltage display.
- 6. Back off the ball screw until the zero point appears on the display.
- 7. Plug data logger USB into computer.
- If this experiment is occurring at 50 °C, allow the specimen to sit in the convection oven for 11 minutes.
- 9. Startup data logger software and begin recording data.
- 10. Wait 1 minute.
- 11. Plug the motor power supply in.
- 12. The experiment will run automatically.
- 13. Wait 1 minute.
- 14. Unplug the motor power supply.
- 15. Stop the data logger recording and export to Excel.

Parts List

| Part | Use | Supplier | Part Number |
|---|------------------------|-----------------|---------------------|
| OMHT34-485 | Stepper motor | Omega | OMHT34-485 |
| Driver ST10-Si | Stepper driver | Omega | ST10-Si |
| 1210AF-10KN-B | Load cell | Interface Force | 1210AF-10KN-B |
| Steel plate | Outside plates | McMaster Carr | 9143K729 |
| Steel plate | Inner plate | McMaster Carr | 9143K734 |
| Bearings | Rod bearings | McMaster Carr | 6627K433 |
| Metal rods | Support rods | McMaster Carr | 8927K11 |
| Ball screw | Ball screw | McMaster Carr | 5966K4 |
| Ball nut | Ball screw | McMaster Carr | 5966K3 |
| End mount | Ball screw | McMaster Carr | 60755K14 |
| Steel square | Compression fixture | McMaster Carr | 9143K25 |
| Sleeve flange | Compression fixture | McMaster Carr | 5448T26 |
| OMPS300A48 | Power supply | Omega | OMPS300A48 |
| Power cord se | Power cord Plunge | Omega | Power cord se |
| Metal rods | compression fixture | McMaster Carr | 1482K42 |
| Flanges mounts | Support rods | McMaster Carr | 62645K42 |
| 72 teeth gear | Drive assembly | SDP-SI | A 6C 7-25B72 |
| 9 teeth gear | Drive assembly | SDP-SI | A 6C 7-25B09 |
| Gear chain OM-CP- | Drive assembly | SDP-SI | A 6Q 7-25 OM-CP- |
| BRIDGE101A- 30MV | Data logger | Omega | BRIDGE101A- 30MV |
| Panel Monitor SGA AC/DC | Voltage display | McMaster Carr | 7681K61 |
| Powered Signal Conditioner | Strain amplifier | Interface Force | SGA/A |
| UL Class RK5 Fuse 2A, 250V AC/125V DC | Fuse | McMaster Carr | 7049K184 |
| Class 8.8 Steel Hex Head Screw Zinc- Plated, M7 X 1 mm Thread, 20 mm Long, Packs of 25 | Fastener | McMaster Carr | 91280A414 |

| Part | Use | Supplier | Part Number |
|---------------------|------------|-----------------|-------------|
| Type 316 Stainless | | | |
| Steel Shim Stock | Shim | McMaster Corr | 2217K50 |
| Sheet, 0.015" | SIIIII | Wiewiaster Call | 231/K39 |
| Thick, 8" X 12" | | | |
| Zinc Yellow- | | | |
| Chromate Plated | | | |
| Hex Head Screw | | | |
| Grade 8 STL, 1/4"- | Fastener | McMaster Carr | 92620A552 |
| 20 Thrd Sz, 2-1/2" | | | |
| Lg, Fully Thrded, | | | |
| Packs of 10 | | | |
| Med-Strength | | | |
| Class 8.8 Steel Hex | | | |
| Head Screw Zinc- | | | |
| Plated, M5 X 0.8 | Fastener | McMaster Carr | 91280A238 |
| mm Thread, 30 | | | |
| mm Long, Packs of | | | |
| 50 | | | |
| Zinc-Plated Steel | | | |
| Hex Nut Low- | | | |
| Strength, M5 X 0.8 | Fastener | McMaster Carr | 90591A146 |
| mm Thread, Packs | | | |
| of 100 | | | |
| Low-Strength Steel | | | |
| Hex Nut Grade 2, | | | |
| Zinc-Plated, 1/4"- | Fastener | McMaster Carr | 90473A029 |
| 20 Thread Size, | | | |
| Packs of 100 | | | |
| Low-Strength | | | |
| Zinc-Plated Steel | | | |
| Hex Head Screw | T . | | 01000 + 540 |
| 1/4"-20 Thread | Fastener | McMaster Carr | 91309A542 |
| Size, 1" Long, | | | |
| Packs of 100 | | | |
| Low-Strength | | | |
| Zinc-Plated Steel | | | |
| Hex Head Screw | | | |
| 1/4"-20 Thread | Fastener | McMaster Carr | 91309A556 |
| Size, 3-1/2" Long. | | | |
| Fully Threaded. | | | |
| Packs of 25 | | | |

| Part | Use | Supplier | Part Number |
|--------------------|-----------------|------------------|-------------|
| Zinc-Galvanized | | | |
| 90 Deg Angle 1- | | | |
| 1/2" X 1-1/2" Leg | s, Flex fixture | McMaster Carr | 8968K61 |
| 1/4" Thickness, 1 | 1 | | |
| Length | | | |
| Ultra Wear- | | | |
| Resistant D2 Too | 1 | | |
| Steel Tight- | Ded | MoMostor Com | 99565V1 |
| Tolerance Rod, | Kou | Inciviaster Carr | 00303K1 |
| 1/8" Diameter, 1/2 | 2' | | |
| Long | | | |
| Low-Carbon Stee | -1 | | |
| Rod 1/2" Diamete | r, Rod | McMaster Carr | 8920K155 |
| 1' Length | | | |
| Fully Threaded T | - | | |
| Slot Nut 1/4"-20 | Eler forteres | MaMastan Com | 04750 4 599 |
| Thread Size, for | Flex fixture | McMaster Carr | 94/30A388 |
| 5/16" Slot Width | l | | |
| Machinable-Bore | | | |
| Clamping Shaft | | | |
| Coupling for 1/2' | Gear assembly | McMaster Carr | 3084K31 |
| X 0.235"-0.500" | | | |
| Shaft Diameter | | | |
| 12L14 Carbon | | | |
| Steel Tight- | | | |
| Tolerance Rod 1/4 | Gear assembly | McMaster Carr | 5227T232 |
| Diameter, 1' | | | |
| Length | | | |
| Machinable-Bore | | | |
| Clamping Shaft | | | |
| Coupling for 7/8 | Gear assembly | McMaster Carr | 3084K47 |
| X 0.235"-0.875" | - | | |
| Shaft Diameter | | | |
| 12L14 Carbon | | | |
| Steel Tight- | | | |
| Tolerance Rod 7/8 | " Gear assembly | McMaster Carr | 5227T312 |
| Diameter, 1' | • | | |
| Length | | | |

| Part | Use | Supplier | Part Number |
|---|---------------|---------------|-------------|
| Machinable-Bore Clamping Shaft Coupling for 7/8" X 0.235"-0.875" Shaft Diameter | Gear assembly | McMaster Carr | 3084K47 |
| Rotary Shaft 1566 Carbon Steel, 1/2" Diameter, 12" Long | Gear assembly | McMaster Carr | 1346K17 |
| Idler Pulley for 7/16" Wide Flat Belt | Chain tensor | McMaster Carr | 1355K1 |
| 18-8 Stainless Steel Oversized Washer for NO. 12 Screw Size, 0.25" ID, 1" OD, Packs | Chain tensor | McMaster Carr | 90313A509 |
| Low-Carbon Steel Bar 2" Thick, 3- 1/2" Wide, 1/2 ft. Long | Chain tensor | McMaster Carr | 8910K106 |

APPENDIX B

Wire Diagram

