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Preparation of Mesophase Pitch Feedstock for Carbon Fiber

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Preparation of Mesophase Pitch Feedstock for Carbon Fiber

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1. LIST OF ACRONYMS

UKY CAER	University of Kentucky, Center for Applied Energy Research
ACP	Advanced Carbon Products
ACP-20	Advanced Carbon Products mesophase pitch
PS	Pressure Spin
SEM	Scanning Electron Microscopy
GPa	Giga Pascal
MSI	Millions of pounds per square inch

1.1 List of Figures

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1.4 Acknowledgements

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2. EXECUTIVE SUMMARY

A filtration process of the low cost mesophase pitch developed by Advanced Carbon Products, LLC (ACP) was developed. The mesophase pitch was melt spun into fiber at the University of Kentucky Center for Applied Energy Research (UKY), and converted to carbon fiber. The melt spinning process was moderately stable, but resulted in some void structure in the green fibers. The tensile properties of the resulting carbon fibers, after graphitization heat treatment, were tested yielding a modulus value of approximately 560 GPa or 81 MSI. This demonstrated that a high modulus graphitic carbon fiber is certainly possible from the ACP 20 mesophase pitch. For future work, a better balance between the softening point temperature, the spinning temperature, and filtering must be met to reduce the volatiles generated in the pitch during spinning. This would reduce voids in the fiber, facilitate spinning, and generate finer, higher performance graphitic fiber.

3. INTRODUCTION

The purpose of this project was to enable the team to learn how to filter feedstocks, isotropic and mesophase pitches to remove impurities that impact the successful melt spinning or melt blowing of pitches. The developed techniques were applicable to processing many different feedstocks and produced pitches including coal and petroleum-based oils and pitches. This knowledge can be used to produce acceptable feedstocks for the production of low-cost carbon fiber.

The study was ~~also~~ intended to determine if the mesophase produced by a novel process developed and patented by ACP can successfully be melt processed into a carbon fiber with the properties required for the automotive industry. Data generated from this study will be used to estimate the cost of producing an ACP pitch based carbon fiber. We anticipate this data will support that a $5/lb carbon fiber could be produced for the automotive industry.

The two ACP patented processes allow isotropic and mesophase pitch to be manufactured at significantly lower capital, operating, and energy costs. An additional benefit of these new processes, is that the carbon footprint for producing mesophase pitch as a feedstock for carbon fiber will be the lowest of all the processes currently being used for producing a carbon fiber feedstock . This project endeavored to demonstrate that this new technology for producing mesophase pitch could be used to produce carbon fiber with acceptable properties and fully productionized cost in the neighborhood of $5/lb.

4. BACKGROUND

Advanced Carbon Products, LLC (ACP) has developed and patented processes for producing isotropic and mesophase pitch (see ACP patents US09222027 & US09376626). The first patent is used to produce a high-quality isotropic pitch from petroleum or other feedstocks. This process is able to use a high-volume low-cost petroleum oil. This is very important because this feedstock is produced every day in quantities that would allow hundreds of thousands of tons of carbon fiber to be produced each year without any feedstock capital investment. Because this is a continuous process, the isotropic pitch can be produced at a low cost. The isotropic pitch then becomes the feedstock used to make mesophase pitch. The second patent covers the production of a low-cost high quality mesophase pitch suitable for spinning into a high-quality carbon fiber. The importance of this patent is that it is the first continuous process for making mesophase pitch. Secondly, this process converts the isotropic pitch into mesophase pitch in less than one second utilizing superheated steam. These two process innovations are the key to producing low cost mesophase pitch. Until these innovations, the state-of-the-art for producing mesophase pitch was in a batch process that required the heating/processing of the isotropic pitch for up to 24 hours. These patents are the key to be able to produce high quality carbon fiber at low costs. Together, these two processes will allow isotropic and mesophase pitch to be manufactured at significantly lower capital, operating, and energy costs. An additional benefit of these new processes is that the carbon footprint for producing mesophase pitch as a feedstock for carbon fiber will be the lowest of all the processes currently being used for producing a carbon fiber feedstock. The purpose of this project was to demonstrate that this new technology for producing mesophase pitch can be used to produce carbon fiber with acceptable properties.

5. RESULTS AND DISCUSSION

Technical approach and hypothesis guiding this approach
ACP had produced mesophase pitch in their pilot plant. The mesophase pitch was determined to be of the quality suitable for spinning into carbon fiber. The issue that needed to be addressed was the solids that remained in the mesophase pitch. The mesophase pitch had been made from a petroleum feedstock. During the processing of petroleum crude oils, small amounts of solids, in this case catalyst particles, get carried through the process and end up in the bottom heavier products. The remaining solids were approximately 0.1 – 0.2 wt. %. Working with UKY CAER, it was determined that the target level of solids should be below 20 ppm. The technical challenge was to remove these solids from a 325°C softening point mesophase pitch. The best approach to remove the solids was to filter them out. ACP then designed and built a filtration system to meet this objective.

The filtered mesophase ACP 20 pitch was received at UKY for spinning trials and conversion to carbon fiber. First the as-received pitch was analyzed for softening point temperature and mesophase content, then post treated by vacuum distillation to remove volatiles. Second, spinning trials utilizing nitrogen over-pressure spinning of the molten pitch were undertaken, until the most suitable spinning conditions were found. Lastly, the spun green fibers were batch-converted to carbon fiber, treated to graphitization temperatures, tested and analyzed.

Experimental methodology, test procedures, characterization methods

Because of the high softening point temperature of the mesophase pitch, direct filtration of the mesophase pitch was not practical. The procedure selected was to dilute/dissolve the mesophase pitch in a solvent. Once diluted/dissolved the material was filtered through a 0.3-micron filter. Based on the particle size of the retained solids, it was determined that this procedure would remove the solids down to the target level of 20 ppm. ACP designed a filtration system that would be capable of removing the solids down to the target level.

The filtration unit had to be designed to be heated to 500°F and hold pressures up to 500 psig. The unit had to be designed to include a mixer in it to maintain dilution of mesophase in the solvent. The bottom of the filtration unit was connected to a housing that would contain the 0.3-micron filter. From the review of open literature, best solvent for dissolving mesophase pitch was found to be quinoline.   This presented several problems from the very strong odor and worker exposure. The entire unit had to be operated under a hood to maintain safety. Once the filtration unit was built, the mesophase pitch and quinoline were placed into the filtration unit and heated. During heat-up the mixer was operated to mix the mesophase pitch and solvent together. Once at temperature, pressure was applied and a valve opened to send the mixture through the filter.

After filtration, the mixture was distilled to remove the quinoline. It was determined that some quinoline remained in the mesophase pitch after distillation. A procedure was then added to place the mesophase pitch in a high temperature vacuum unit to remove the remaining quinoline. The sample was then shipped to UKY CAER.

The as-received ACP 20 mesophase pitch was examined for softening point temperature by dynamical mechanical analysis (DMA) under a nitrogen purge. Here a 1/8" diameter disc of pressed pitch powder was loaded in compression under a constant load of 0.1 N and a heating rate of 3 ºC/min. The percent mesophase was determined using ASTM D4616-95 methodology by point counting of regions of optical anisotropy observed in photomicrographs by polarized reflected light microscopy. A sample of pitch was embedded in epoxy and metallographically polished for the imaging process.

Prior to spinning, the pitch was subjected to a vacuum distillation process wherein it was heated to 325 ºC in an initial vacuum < 1 Torr for 10 min. The pitch spent approximately 40 min total above 300 ºC. The vacuum distillation process is described in more detail in Appendix B.

Spinning trials utilized a temperature-controlled pressure vessel with an attached single hole spinneret, in which a sintered stainless steel frit was press fit. This allowed for temperature control of the pitch, nitrogen over-pressure to force out the molten pitch, and for the molten pitch to pass through a 40 micron sintered frit prior to entry into the spinneret capillary. The emerging pitch extrudate was drawn down and fixed to the surface of a rotating spool, which spun the green fiber, and collected on its surface. The best spinning conditions were as follows:

Vacuum Distilled Sample:	PS174-1
Spinneret diameter:	330 μm
Stainless Steel Frit:	40 μm
Spin Temperature:	343-347 ºC
Nitrogen Over-Pressure:	100-200 psi
Spool Speed (3.25" OD):	55 m/min

The green fiber was cut from the spool as a collimated bundle and batch processed through oxidation, carbonization and graphitization. Given the high Tsp of the pitch, no observance of inter-filament fusion, post oxidation processing, was observed. This process ultimately generated carbon fiber with an average diameter of 34.8 +/- 5.8 micron.

The carbon fiber was tested by single filament tensile tests using a MTS QTest 10 materials testing machine with filaments mounted to aperture cards with epoxy at gauge lengths of 20, 30, 40 and 50 mm (ASTM D3379-75). However, each individual filament diameter was determined by a Keyence laser measurement system, prior to each test.

Presentation and discussion of results

The final filtered mesophase pitch was tested at ACP for softening point (Mettler), mesophase content (Optical microscopy via computer analysis) and carbon content (MCRT). The filter element was examined to quantify how effective the filtration had been. It was determined that the majority of the solids had been removed, but the exact amount could not be quantified. The ash from the MCRT analysis was examined for remaining solids. The examination indicated that the remaining solids in the mesophase pitch was very low, but could not be quantified. Our analytical capability was not capable of getting down to the ppm level. This was identified as an element that would need to be developed in future work.

The deformation of the pitch in the DMA as a function of temperature was recorded, and its first derivative with respect to temperature analyzed. The peak of this curve corresponded to the temperature at which the pitch was deforming fastest, and recorded as the softening point temperature, Tsp, at approximately 274 to 292 ºC. The pitch did display a bit of a bimodal Tsp character as shown in Figure 1.

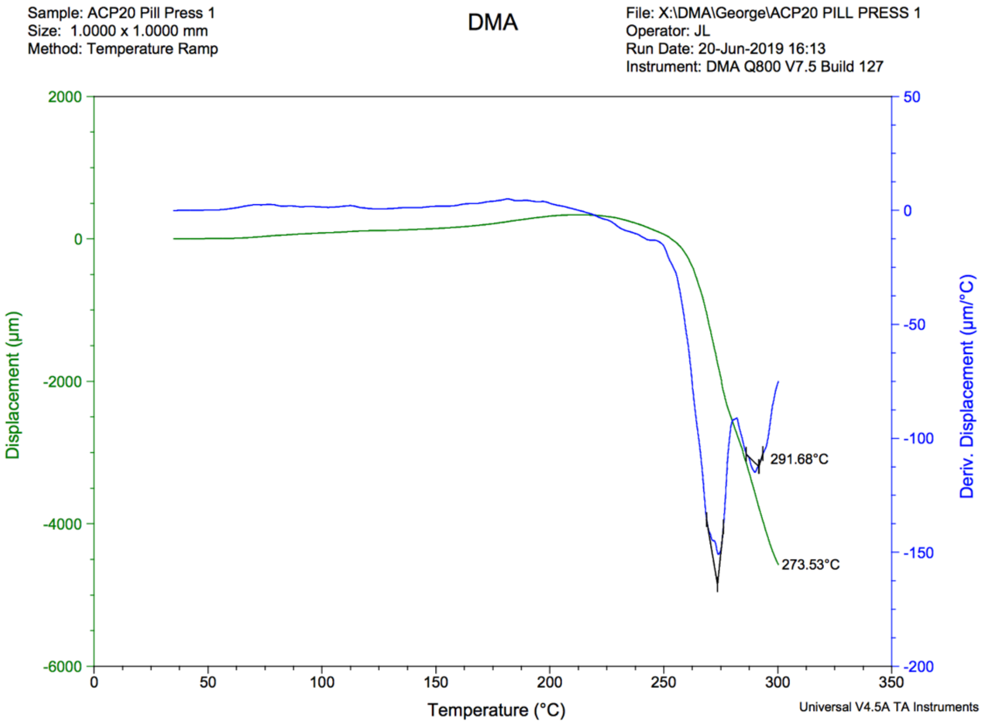


Figure 1. Softening point temperature of the as-received ACP 20 mesophase pitch material

The point counting of the photomicrographs indicated the ACP 20 was 95% mesophase (optically anisotropic) material. The images in Figure 2 are representative of the material.

Initial spinning trials suggested early on that the filtered ACP 20 likely had residual volatile material in it, as indicated by large void structures observed in the green filaments (Figure 3). Therefore, prior to spinning, the pitch was subject to the vacuum distillation process. Only approximately 0.4 wt.% of the material was lost to vapor in the process, but the resultant fibers showed less void structure. Still, small voids were observed in the green fibers, particularly near the filament surface, consistent with some sort of off-gassing phenomena.

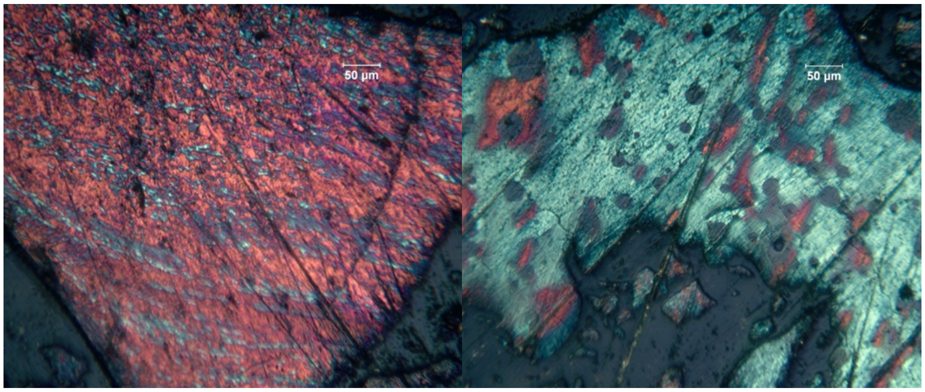


Figure 2. Polarized reflected light microscopy of the ACP-20 mesophase pitch

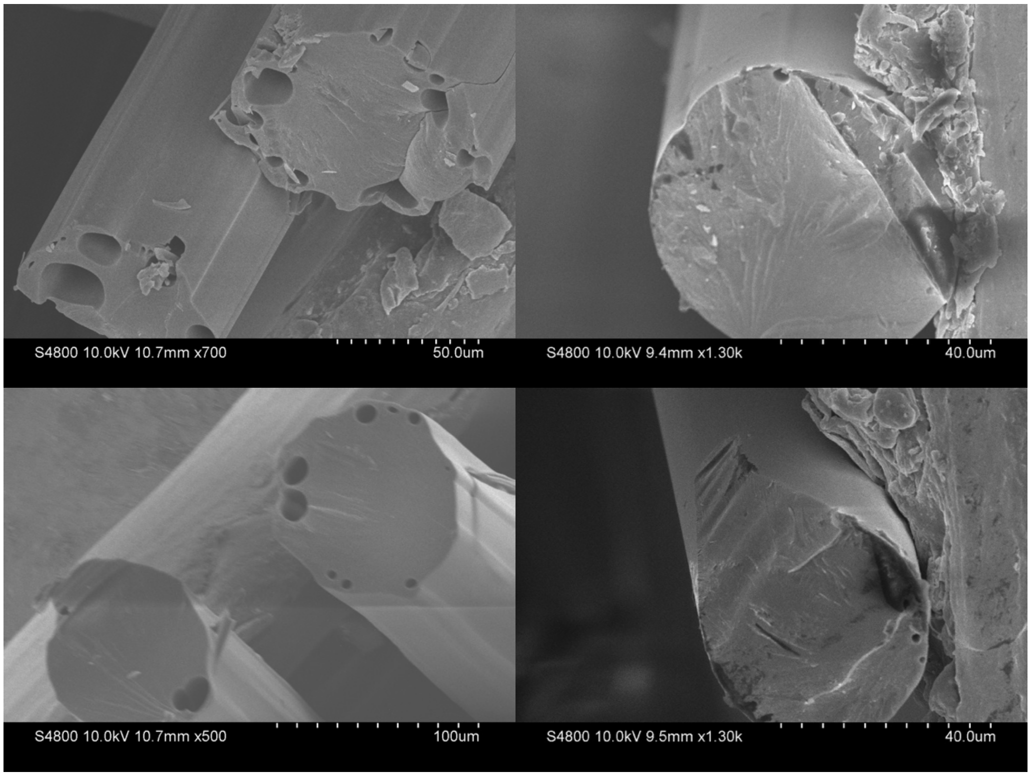


Figure 3 (left column – PS 173-1) SEM images of green ACP 20 fiber without vacuum distillation, and (right column – PS 174-1) with vacuum distillation.

The spinning process was challenging overall. The pitch behaved quite viscous and resistant to flow. In an effort to generate smaller diameter fiber, a 150 micron diameter capillary was attempted, but nearly no pitch extrudate was attained. At increased temperatures, the pitch appeared to react and become even less amenable to extensible flow. The spool take up speed reported above represented the apparent maximum, for relatively stable draw down achievable for this pitch. These issues, complicated with the tendency for the material to off-gas, limited the fineness of the final carbon fibers. Nonetheless, we persisted in converting and analyzing the resultant graphitic carbon fibers.

Figure 4 shows the fracture surfaces of the carbon fibers generated from the ACP 20 mesophase pitch (after graphitization processing). Graphitization was done by heating the carbonized filaments in a flowing He atmosphere, 50 ºC/min to temperatures > 2200 ºC and held for 10s of minutes. At such temperatures, axially-aligned crystalline graphite domains form from mesophase-based fibers. Clearly significant void structure persisted in the filaments through the thermal conversion processing (Figure 4). Moreover, some void structure may have stemmed from any residual FCC catalyst/alumina support particles, that remained in the pitch. However, the graphitic texture, and semi-radial stacking of the graphitic planes was clear. This observation indicated that the fiber modulus should be quite high, but the voids would likely act as stress concentrations and reduce the tensile strength significantly. Also, the filament surfaces appeared to be rather smooth, apart from linear micro-striations along the fiber length.

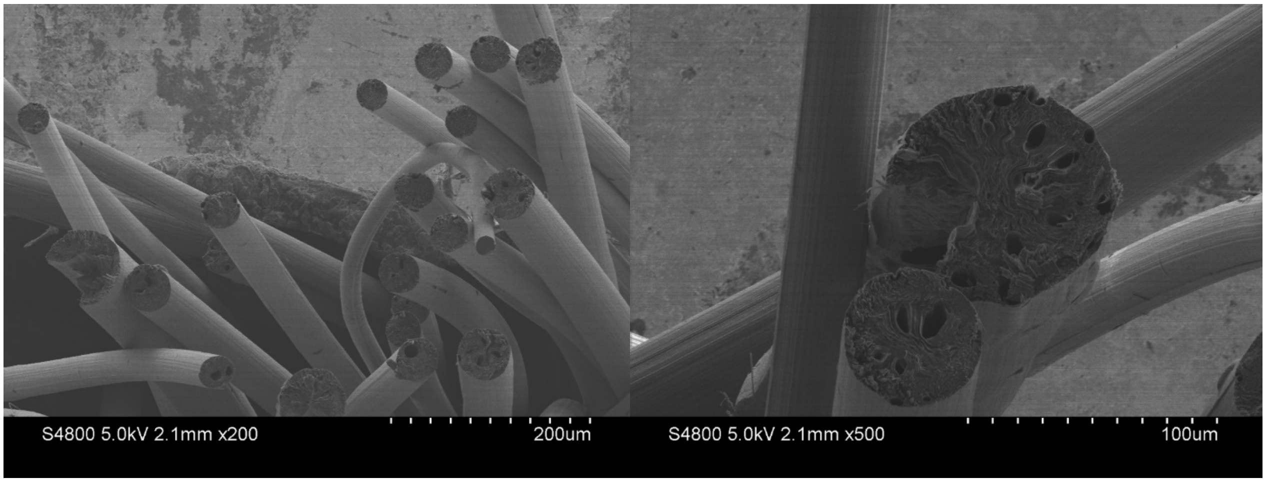


Figure 4. SEM images of fracture surfaces of the graphitized fiber from ACP 20 mesophase.

The final carbon fiber tensile properties were determined by single filament tensile testing. The carbon fiber was immensely brittle, consistent with both the graphitic texture observed, and magnified by the omnipresent macro voids throughout the fibers. Of the 146 filaments tested (25 to 50 at each gauge length), only 38 tests were considered representative of the fiber (e.g. free of filament breakage upon mounting, breakage at the glue, pre-tension failures, etc.). At the gauge lengths of 20, 30, 40 and 50 mm; 6, 13, 10 and 9 filaments were successfully tested respectively. A summary of the results are given in Table 1 below.

Table 1. Carbon Fiber Properties

					Corrected		Corrected		Corrected		
Gauge Length (mm)	Diameter (micron)	standard deviation (micron)	Stress At Break (MPa)	standard deviation (MPa)	Modulus (GPa)	standard deviation (GPa)	Strain at Break (%)	standard deviation (%)	Strain Energy Density (MJ/m3)	standard deviation (MJ/m3)	N
20-50	34.84	5.82	465.00	308.14	560.05	176.22	0.077%	0.04%	0.24	0.23	38

After the system compliance correction was applied (see Appendix B), the modulus was 560 GPa (or 81 MSI), well higher than the targeted 25 MSI. System compliance correction effectively converts the recorded crosshead displacement into strain in the fiber gauge length. During tensile testing of a high modulus fiber using a delicate load cell, small displacements stemming from load cell extension can be quite similar to the actual displacements in the fiber. The system compliance correction accounts for these by extrapolating measured displacements (crosshead displacements) to a zero gauge length fiber. In esscence, fiber tensile testing is like two springs in series – one is the fiber, and the other the load cell (system). This correction allows for the determination of the spring constant (and later, modulus) of the fiber, by quantiatively determining the spring constant of the load cell (system). More detail is given in Appendix B.

The strength and strain at break were quite low at 465 MPa (67 ksi) and 0.077 % respectively. However, as the modulus of the fiber was observed to be 81 MSI, one can conclude that a high modulus graphitic carbon fiber is certainly possible from the ACP 20 pitch.

For future: A better balance between the softening point temperature, the spinning temperature, and filtering must be met to reduce the volatiles generated in the pitch during spinning and processing. This would reduce voids in the fiber, facilitate spinning, and generate finer, higher performance graphite fiber.

6. BENEFITS ASSESSMENT

Review of ACP's pitch processes indicated significant benefits in several key areas:

1. The developed processes are continuous and capable of producing large quantities of isotropic and mesophase pitch at low costs.
2. The use of a continuous process reduces the energy requirement to produce one pound of pitch.
3. The processes developed by ACP has very low environmental emissions.
4. The carbon footprint of the ACP processes are significantly lower than that of PAN.
5. The processes utilize feedstocks that are currently being produced today in high volume which are capable of producing tens of thousands of tons of carbon fiber. In contrast, the expansion of PAN would require construction/expansion of raw materials used to make PAN.
6. The cost of building and operating a mesophase pitch carbon fiber can be between 30% to 50% lower than a PAN based carbon fiber plant.
7. All of these benefits and lower costs should allow for carbon fiber to be produced at a low cost.

7. COMMERCIALIZATION

The project has confirmed that a carbon fiber can be produced from the mesophase pitch supplied from ACP. Additional work has been indicated, in this study, to improve the quality of the produced carbon fiber. This work should help prepare for the commercialization of these processes.

ACP has been scaling up the pilot plants to produce the mesophase, which should be completed by the end of 2019. This work will demonstrate scalability of the processes and establish operating parameters. The processes will then be ready for commercialization.

8. ACCOMPLISHMENTS

Summarize the project accomplishments in relation to the project objectives including process/product development, technology transfer, and commercialization activities. It can include a listing of:

i) Any awards (such as R&D 100) received
ii) Publications in scientific/ trade journals, conference proceedings etc
iii) Patents – granted, applications, or disclosures; licensing agreements, etc.
iv) Graduate students thesis based on the project work
v) Web site or other Internet sites that reflect the results of this project
vi) Other products (e.g. software, data bases, inventions

None of the above.

9. CONCLUSIONS

The project developed a filtration methodology for mesophase pitch to reduce its solids content to 20 ppm. Even though the target solids content was not achieved, several strategies were proposed for follow on development. The recommendation of the team is that solids removal be completed before the making of the mesophase pitch. This procedure will be incorporated in any future projects in making carbon fiber. The project confirmed that a mesophase pitch produced by a continuous process can produce a produce a prototype carbon fiber, which was observed to have a high modulus (81 MSI). As identified below, additional work should be conducted to facilitate the production of a high-quality carbon fiber.

We learned from this project that a better balance between the softening point temperature, the spinning temperature, and filtering must be met to reduce the volatiles generated in the pitch during spinning and processing. This would reduce voids in the fiber, facilitate spinning, and generate finer, higher performance graphite fiber.

10. RECOMMENDATIONS

1. Removal of all solids should be accomplished before the production of the mesophase pitch.
2. Additional work should be performed to improve the softening point of the mesophase pitch to address volatiles generated during the spinning process and to improve drawability to reduce the diameter of the carbon fiber.
3. Additional work should be performed to improve the overall quality of the produced carbon fiber.
4. Work should also be conducted in the areas of stabilization, carbonization and graphitization to not only improve the carbon fiber quality, but to also reduce manufacturing cost.

11. REFERENCES AND/OR BIBLIOGRAPHY

None referenced.

12. APPENDICES

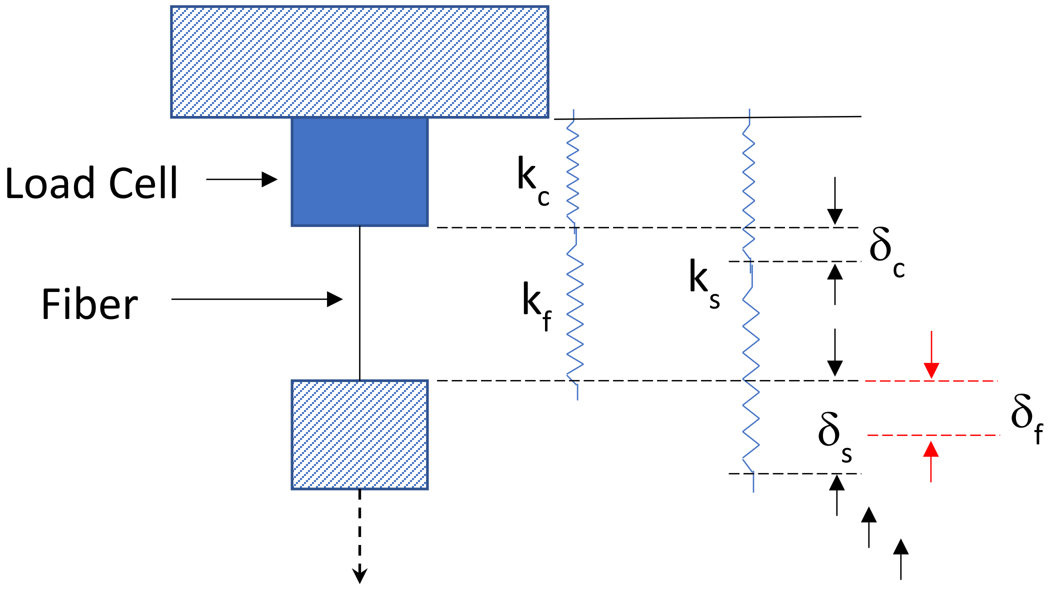
APPENDIX A: Vacuum Distillation Of The Pitch

Remember: Wear proper safety equipment (goggles, gloves, lab coat (recommended)) and wash hands after setup is complete.

1. Weigh out the desired amount of material (nominally 500 g) into the 2000 mL jar, then record the weight.
2. Setup the glassware, thermocouples, data logger, stirring rod, and vacuum pump.
   a. Ensure the pitch thermocouple is as straight as possible and extends as far into the jar as possible without contacting the side or bottom.
   b. Ensure that both of the mantle thermocouples are close together and taped at the bottom of the mantle. (This will eliminate as much variance as possible between the data logger and the mantle temperature controller)
3. Turn the vacuum pump on and test the system for any leaks. The vacuum inside the setup should reach the millitorr range to be deemed ready for vacuum distillation purposes.
4. Set the pitch temperature controller (TC) to approximately 40°C below the target temperature. (This will ensure that the pitch temperature does not overshoot the target temperature)
   a. Turn on stirring motor if needed.
5. Slowly increase the pitch temperature until the target temperature is reached.
6. Hold at target temperature for desired amount of time with vacuum pump running.
7. After desired time at temperature has passed turn off pitch temperature controller and allow system to cool.
   a. Keep the vacuum pump running during the entire cool down.
   b. Keep the stirring motor running until the pitch temperature has dropped approximately 30-40°C.
8. Turn off the vacuum pump once the system has completely cooled.

APPENDIX B: System Compliance Calculation for the Tensile Modulus of the Carbon Fiber

Given that the tensile testing of high modulus carbon fibers is effectively 2 springs in series (one is the fiber, the other is the load cell & ‘system’), and that deformations in the carbon fiber are on the same lengthscale as those in the ‘system’, one needs to correct the data to determine the actual Tensile or Young’s modulus of the fiber, as well as its break strain.



$$\delta_s = \delta_c + \delta_s$$

$$k_s = \frac{\vec{F}}{\delta_s} \quad k_c = \frac{\vec{F}}{\delta_c} \quad k_f = \frac{\vec{F}}{\delta_f}$$

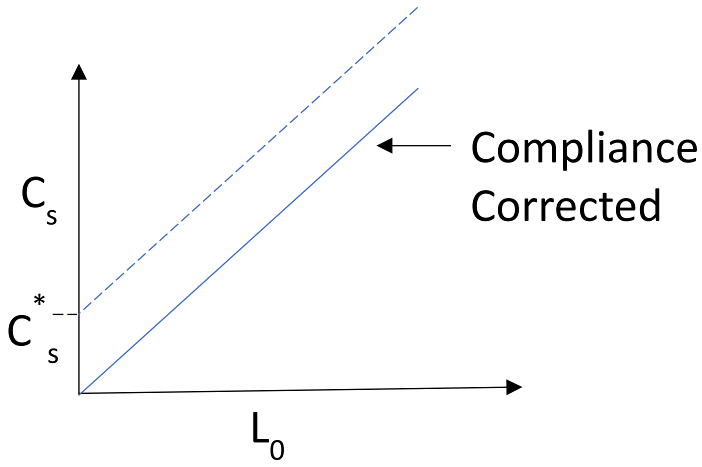
For a single fiber in tension, the overall displacement δ_s, is the sum of the displacements in the load cell (and glue, etc.) and fiber, δ_c and δ_f respectively (subscripts s, c, and f represent system, load cell, and fiber respectively). This is essentially two springs in series (with stiffnesses k), and the force F is constant through both. In normal testing conditions the stiffness of the load cell, k_c, is much greater than that of a single fiber, k_f. However for soft load cells (needed for testing single filaments), combined with high modulus fiber, the concern is that as the gauge length of the fiber gets small then $\delta_f \approx \delta_c$. This causes large errors in the measurement of the fiber modulus. However, at zero gauge length, we would theoretically measure only the displacement of the load cell. Once we have this, we can correct the measurements to give us the true strain in the fiber, and its Young’s modulus.

First, we must extrapolate the measured total compliances, C_s, to zero gauge length.

$$C_s = \frac{1}{k_s} = \frac{\delta_s}{\vec{F}} \qquad \sigma = \frac{\vec{F}}{A} \qquad \varepsilon_s = \frac{\delta_s}{l_0} \quad E_s = \frac{\sigma}{\varepsilon_s}$$

A is the fiber cross sectional area, l_0 its gauge length, and σ the stress in the fiber. E_s is the apparent system modulus, ε_s is the apparent measured strain of the system.

The y-intercept of this graph is the **system compliance** C^*_s, and is then subtracted from all the other measured compliances to correct them for displacements in the load cell.



The measured modulus of the two spring system:

$$E_s = \frac{\vec{F}/A}{\delta_s/l_0} = \frac{\vec{F} l_0}{A\delta_s} = k_s\left(\frac{l_0}{A}\right)$$

With

$$k_s = \frac{\vec{F}}{\delta_s} \quad and \quad \frac{1}{k_s} = C_s$$

So

$$\frac{1}{E_s} = C_s\left(\frac{A}{l_0}\right)$$

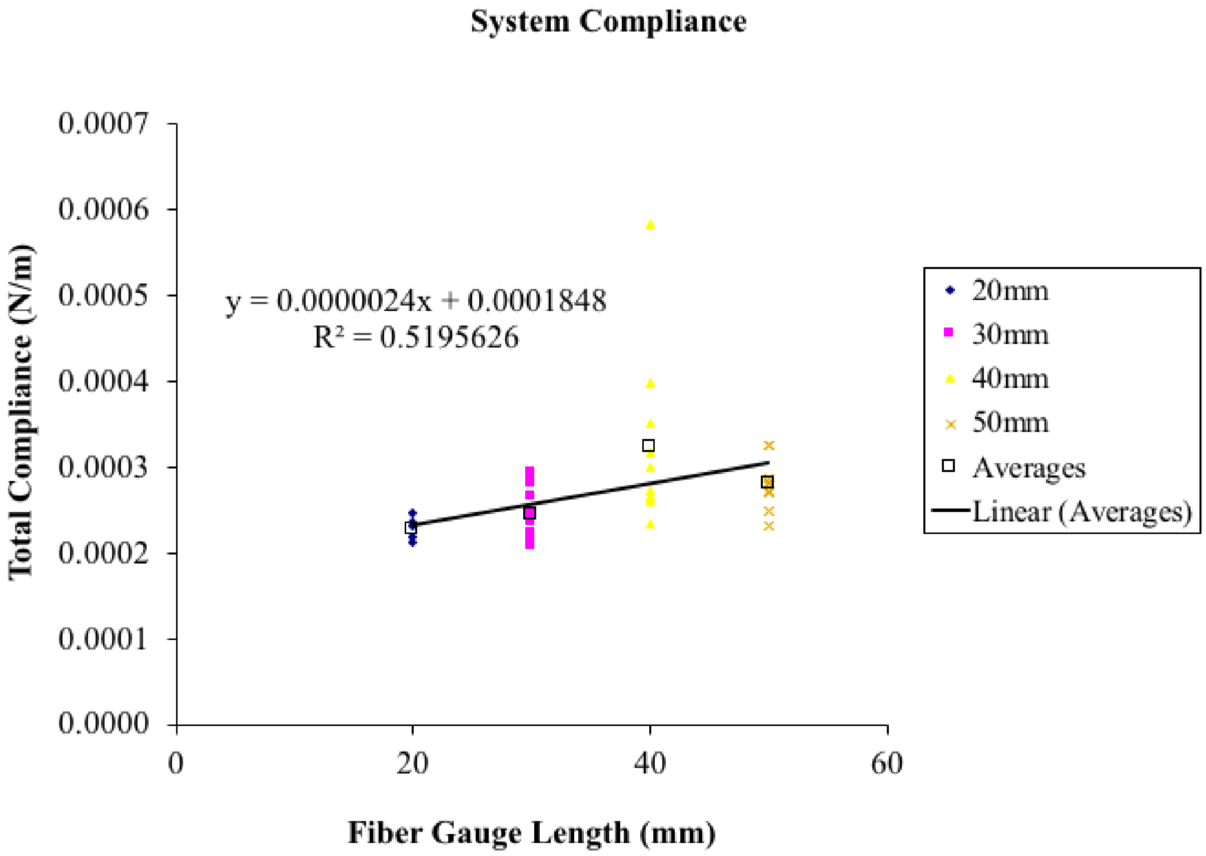
And

$$C_s = \left(\frac{l_0}{A}\right)\left(\frac{1}{E_s}\right)$$

So the corrected Young's modulus of the fiber is then:

$$E_Y = \left(\frac{l_0}{A}\right)\left(\frac{1}{C_s - C_s^*}\right)$$

We plotted the total compliance as a function of gauge length, and extrapolated to zero gauge length to get the 'system compliance'. The y-intercept, or system compliance was 0.0001848 N/m, which was then used to correct the apparent moduli through the above formula. The R^2 was quite low, and consistent with the difficult testing of these brittle fibers.


System Compliance
Total Compliance (N/m)
0.0007
0.0006
0.0005
0.0004
0.0003
0.0002
0.0001
0.0000
y = 0.0000024x + 0.0001848
R² = 0.5195626
0
20
40
60
Fiber Gauge Length (mm)
20mm
30mm
40mm
50mm
Averages
Linear (Averages)

APPENDIX C: Carbon fiber single filament tensile testing results

					Corrected		Corrected		Corrected		
Gauge Length (mm)	Diameter (∝m)	std. dev. (∝m)	Stress At Break (MPa)	std. dev. (MPa)	Modulus (GPa)	std. dev. (GPa)	Strain at Break (%)	std. dev. (%)	Strain Energy Density (MJ/m3)	std. dev. (MJ/m^3)	N
20-50	34.84	5.82	465	308	560	176	0.08%	0.04%	0.24	0.23	38
20	35.0		463		634		0.07%		0.17		
20	37.0		980		664		0.15%		0.72		
20	33.0		198		377		0.05%		0.05		
20	35.0		151		408		0.04%		0.03		
20	34.0		684		479		0.14%		0.49		
20	34.0		872		729		0.12%		0.52		
30	45.0		661		604		0.11%		0.36		
30	40.0		75		295		0.03%		0.01		
30	45.0		613		635		0.10%		0.30		
30	40.0		283		634		0.04%		0.06		
30	39.0		69		265		0.03%		0.01		
30	43.0		656		670		0.10%		0.32		
30	49.0		565		616		0.09%		0.26		
30	27.6		702		476		0.15%		0.52		
30	32.4		197		597		0.03%		0.03		
30	27.0		609		483		0.13%		0.38		
30	28.0		198		509		0.04%		0.04		
30	41.5		785		934		0.08%		0.33		
30	31.5		980		753		0.13%		0.64		
40	30.0		347		718		0.05%		0.08		
40	31.0		102		405		0.03%		0.01		
40	24.0		160		222		0.07%		0.06		
40	39.0		606		703		0.09%		0.26		
40	26.0		1017		662		0.15%		0.78		
40	34.0		193		502		0.04%		0.04		
40	29.5		92		274		0.03%		0.02		
40	31.5		634		690		0.09%		0.29		
40	34.5		127		257		0.05%		0.03		
40	29.0		1050		760		0.14%		0.72		
50	36.5		137		340		0.04%		0.03		
50	37.0		357		477		0.07%		0.13		
50	30.0		820		738		0.11%		0.46		
50	40.5		286		615		0.05%		0.07		
50	37.0		275		456		0.06%		0.08		
50	31.0		440		762		0.06%		0.13		
50	29.0		194		540		0.04%		0.03		
50	40.0		837		860		0.10%		0.41		
50	37.5		257		538		0.05%		0.06		

APPENDIX D: Quick Review of Project Milestones

Project Milestones

- Milestone 6.18.2.1 Designed and built filtration unit was pressure tested to 2000 psi and held for 1 hour then sealed then the left overnight with no more than 5-10 psi pressure drop. Filtration unit deemed ready for use. (July 2018) (ACP)

- Milestone 6.18.3.1 Mesophase pitch (500 grams), containing ≤ 20 ppm of solid contaminants produced and delivered to UKY. (October 2018) (ACP)

- Milestone 6.18.4.1 Demonstrate at least 3 minutes of continuous melt spinning stability (record process on video) (January 2019) (UKy)

    - 100% Done May 2019

- Milestone 6.18.3.1 Demonstrate ACP mesophase pitch carbon fiber with an average modulus of at least 25 MSI, N > 25 by single filament tensile testing. (April 2019) (UKy)

    - 100% Done June 2019

- Go/No-Go 6.18.1 Demonstrate ACP mesophase pitch carbon fiber with an average modulus of at least 25 MSI, N > 100 by single filament tensile testing. (June 2019) (UKy)
    - 100% Done June 2019