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Effect of Process Variables on Mechanical and Transport Properties of Carbon Fibers from Mesophase Pitch

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INTRODUCTION

- Mesophase pitch is an attractive precursor for carbon fibers due to the potential low-cost of the raw material, the high carbon yield, and the high electrical and thermal conductivities that can be achieved in MP-based carbon fibers [Edie and Dunham, 1989; Diefendorf, 2000; Jeon et al., 2013]
- However, large-scale structural applications of MP-based carbon fibers are limited due to their poor mechanical properties, so controlling the microstructure in the precursor fiber can lead to improved mechanical and transport properties in the MP-based carbon fiber [Endo, 1988; Emmerich, 2014]
- Studies on the relationship of carbon fiber transverse microstructure and carbon fiber properties have been previously reported [Mochida et al., 1996; Edie, 1998]
- However, no important studies have been made on the relationship between draw-down ratio (DDR) and carbon fiber microstructures and properties; something that is understood for the case of melt spinning of polymers [El-Dessouky et al., 2010]

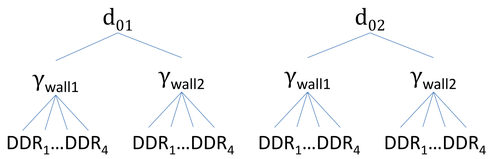
OBJECTIVE

To obtain mesophase pitch-based carbon fibers with improved mechanical strength while retaining superior electrical and thermal properties by

(i) Systematic investigation of the relationship between DDR versus longitudinal and transverse microstructure of mesophase MP fibers; and

(ii) Developing a novel type of microstructure

EXPERIMENTAL DESIGN



PRELIMINARY RESULTS

Spinneret configuration: 12 holes, d_0 = 150 µm

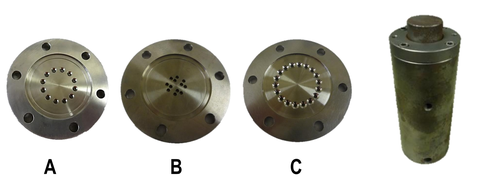
Replicate	1	2	3
Obtained as-spun diameter (µm)	21.8 ± 1.4	19.3 ± 1.7	20.7 ± 1.4
DDR (V_L/V_o)	77	77	76

Achieved consistency in melt spinning by obtaining a repeatable as-spun diameter from a same spinneret and a same DDR.

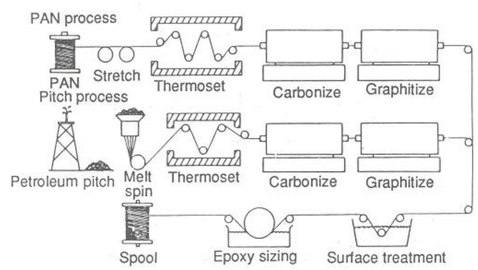
Diameters are not significantly different, with a 99% level of confidence

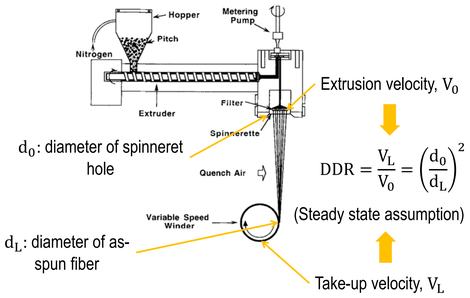
SPINNERET GEOMETRIES

d_0 (µm)	L (µm)	# holes
50	250	18
75	75	12
100	200	12
150	300	12



Available spinnerets for the melt spinning experiments. A: d_0 = 150 µm, 12 holes; B: d_0 = 100 µm, 12 holes; C: d_0 = 50 µm, 18 holes.





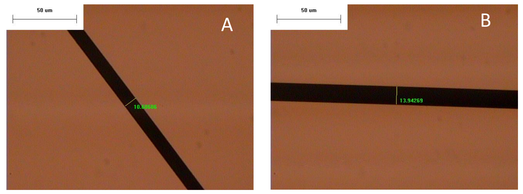
Carbon fiber production using PAN and pitch processes [Diefendorf, 1987; Edie, 1998]

MELT SPINNING OF MESOPHASE PITCH

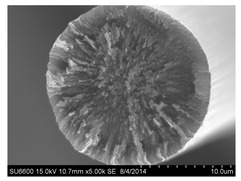
Left: Alex James® bicomponent spinning device. Right : Alex James® batch melt spinning device.

METHODOLOGY

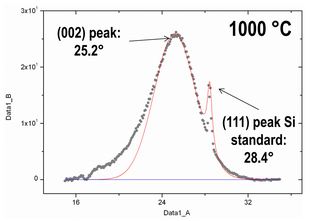
- Melt spinning of mesophase pitch at various draw-down ratios, for different levels of as-spun fiber diameter and shear rate at the capillary wall.
- Stabilization of precursor fibers in air at 200 – 300 °C
- Carbonization and graphitization of stabilized fibers at 2400 °C
- Structural characterization of carbon fibers by WAXD, optical microscopy, SEM and TEM.
- Measurement of mechanical properties by tensile and compressive testing
- Analysis of transport properties by electrical conductivity and thermal conductivity.

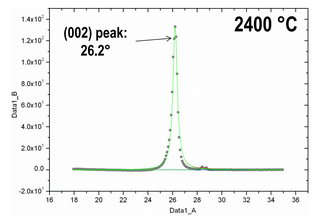


Left: as-spun, mesophase pitch fibers. Middle and right: measurement of as-spun fiber diameter by optical microscopy; specimen A was melt extruded at DDR = 46; specimen B was melt extruded at DDR = 25.



SEM micrograph of AR mesophase pitch-based carbon fiber, showing a radial transverse texture.





WAXD diffractograms for AR mesophase pitch-based carbon fibers. The higher carbonization temperature (2400 °C vs. 1000 °C) leads to a higher degree of graphitic crystallinity as shown by a sharper peak at a larger two-theta.

Left: Netzsch LFA (Laser Flash Analysis) 447 for thermal conductivity measurement. Right: Phoenix MTI universal testing machine for tensile testing and compressive testing.

CONCLUDING REMARKS AND FUTURE WORK

- As-spun fibers with a consistent diameter can be obtained from an AR mesophase pitch using the batch melt spinning device
- The radial transverse texture of AR mesophase pitch-based carbon fibers, analyzed by WAXD and SEM, is consistent with the typical features reported in prior studies
- Mechanical and transport properties will be measured for carbon fibers of same diameter but obtained from different spinning conditions

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