CHARACTERISTICS OF MELT-BLOW SPUN, SOLVATED MESOPHASE PITCH-BASED CARBON FIBER AND TYPICAL COMPOSITES

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Introduction

Potentially low-cost carbon fibers and their composites have been produced and characterized [1]. Solvated mesophase pitch was precursor and a highly productive, patented melt-blow technology for spinning pitch fibers was used[2]. Some unusual characteristic appeared in the carbon fiber processes (including fiber spinning, stabilization, and carbonization and surface treatments) and in the composites. The objective of this study is to systematically investigate the fundamental characteristic of the novel carbon fibers and their composites.

Experimental

Materials: Solvated mesophase pitch from ConocoPhillips Co. was used as raw materials for spinning pitch (green) fibers. Epoxy resin 105 and slow hardener 206 produced by West System, were employed to fabricate composites.

Process for Making Carbon Fibers: Mesophase pitch fibers were produced in the UTSI spin Lab with a high-speed melt blown process. Four different green fibers were spun with blowing flow rates of 40, 60, 80, and 100 Liter Per Minute (lpm), respectively. The green fibers were stabilized in air and carbonized in N_2 in a tube furnace. The process conditions used for stabilization are listed in Table 1.

Table 1. Parameters for Stabilization Process

CF Sample	Stabilization		
	Method	Final temp. (°C)	Level
N-1	Normal	350	Low
2S-2	2 step	250, 350	↓
2S-3	2 step	250, 350	
2S-4	2 step	250, 350	
2S-5	2 step	250, 350	
4S-2	4 step	250, 300 350, 400	High
L-6	Long time	350	

Composite Fabrication: Vacuum bagging resin infusion technique plus a hot press was used to prepare carbon fiber composites. Several layers of carbon fiber were stacked up inside the space between two molds before epoxy resin with hardener enter the system in the bag under vacuum [3].

Characterization: The tensile properties and diameter of single carbon fiber were measured with a Diastron Limited FDAS 765 fiber analyzer that consists of a high resolution tensile tester LEX810 with a force resolution of 0.005 gram and a Mitutoyo LSM-500 Laser Scan Micrometer.

The flexural properties of the carbon fiber composites were tested in an MTS machine with a 550 kN load cell and a head speed of 0.1 inch per minute. A precision extensometer was used to measure strain. ASTM D 6272 standard was used as guide for the testing and calculations.

Results and Discussion

1. Carbon Yield

Four pitch fibers spun using different blowing speeds were stabilized (N-1) and carbonized at same conditions. Fibers yield is shown in Fig. 1. Comparing with pitch fibers, stabilized fibers show < 5wt%; carbonized fibers show ~ 20wt% at 600°C and ~ 25 wt% at 1050°C weight loss, respectively. This indicates that such pitch fibers have a high carbon yield for making carbon fibers.

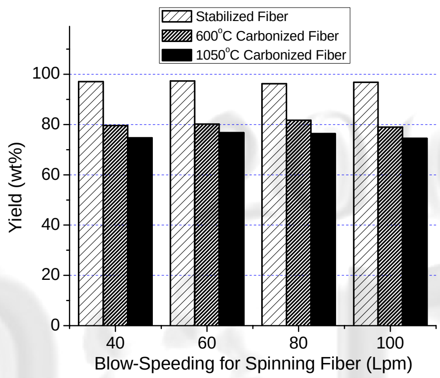


Fig. 1. Yields of fibers after stabilization and carbonization at lower (600°C) and higher (1050°C) temperatures.

2. *Carbon Fiber Diameter*

For the same spinneret used, the diameter of pitch fibers decreased with increasing blowing speeds. After thermal treatment, the diameter of the resultant carbon fibers could be from 10 to 25 μm as shown in Fig. 2. These could be reduced to 7 to 20 μm, if fibers were processed for higher level of stabilization.

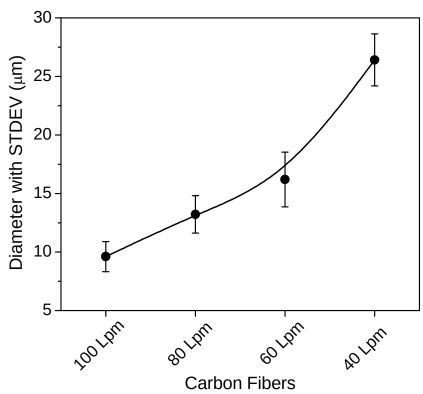


Fig. 2. Diameter of carbon fibers

3. Effect of Stabilization on Tensile Properties

Pitch fiber spun with 40 lpm was used in this study. The fiber was stabilized at different levels as listed in Table 1. After carbonization at 1050°C, the fibers show an increase in tensile strength and modulus (Fig.3) with increasing the level of stabilization. However, it appears that higher level of stabilization will decrease strength and modulus.

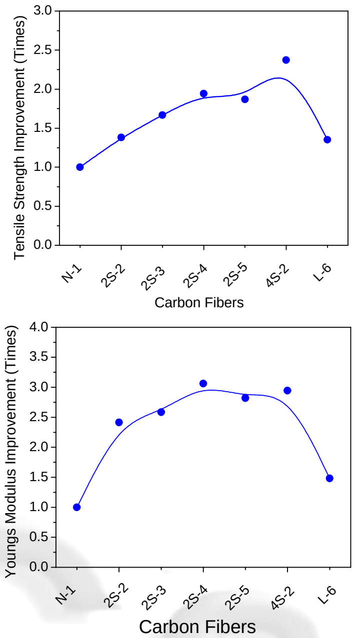


Fig. 3. Tensile strength and modulus of carbon fibers (40 lpm) which suffered different levels of stabilization

4. Effect of Carbonization on Tensile Properties

Fiber tensile properties strongly depend on the carbonization final temperature. At 1050°C, tensile strength and modulus of the fiber (20 -10 μm) can reach 500-1100 Mpa and 70-110 Gpa, respectively. When the carbonization temperature is up to 1500°C, the strength and modulus can be up to ~2.5 Gpa and ~200 Gpa respectively.

5. *Carbon Fiber Form*

As a low-cost process, a typical fiber form made from melt blow technology is a strip of non-woven fabric, about 7.5 cm wide, as shown in Fig. 4. Continuous fibers with many bends are randomly laid down. Even though the laydown process can be designed for the fibers to become significantly aligned.

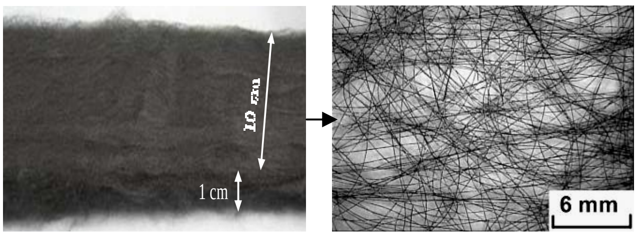
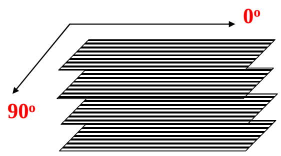


Fig. 4. Typical carbon fiber form

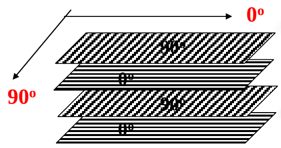
6. *Characteristic of Carbon Fiber Composites*

It is well known that composites made with unidirectional (aligned) carbon fiber have much different mechanical properties along the direction of 0° or 90° with the fiber axis. In this study, our prepared carbon fiber tape (see Fig.4) was fabricated into composites with epoxy resin in two ways as shown in Table 2. Since the fibers were almost randomly aligned, the flexural properties of the composite (top) didn't differ much at 0° and 90°. In the case of multidirectional composites (bottom), the flexural properties are more uniform in two directions.

Table 2. Effect of Fiber Tape Alignment on the Flexural Properties of the Composites.



	Flexural strength (Mpa)	Flexural modulus (Gpa)
0°	184.7	15.1
90°	160.3	11.6



	Flexural strength (Mpa)	Flexural modulus (Gpa)
0°	181.4	15.0
90°	174.6	13.7

Conclusions

Melt-blow spun, solvated mesophase pitch-based carbon fibers have ~ 75 wt% carbon yield at 1050°C. The diameter of carbon fiber was mainly controlled by blowing speed and ranges from 8 to 25 μm. The tensile properties of carbon fibers are strongly influenced by stabilization level and carbonization temperatures. The form of the carbon fiber is different from that of typical commercial products and is in tape shape or non-woven fabric. A single fiber (filament) is continuous long but curved and almost randomly oriented. Using such carbon fibers as reinforcement, the flexural properties of the composite did not display much difference at 0° and 90° of fiber tape direction.

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