PREPARATION OF HOLLOW PITCH-BASED CARBON FIBERS

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Introduction

Properties of pitch-based carbon fibers have been considered could be improved dramatically at little cost, if novel process techniques, such as modifying the fiber shape and structure, are employed [1,2]. Non-circular, especially hollow, pitch-based carbon fibers were proved to have higher mechanical properties than round solid ones because of the improvement of molecular orientation during the spinning [3]. However, an appropriate spinneret has to be used for the precursor pitch with certain rheological properties to spin uniform and continuous non-circular pitch fibers.

In the present work, mesophase and isotropic pitch fibers were spun through C-shaped and triple-arc-shaped spinnerets, respectively, according to the apparent viscosity of the pitches.

Experimental

A mesophase pitch (MP-1) and an isotropic pitch (IP-1), as described in Table 1, were used to spin hollow pitch fibers. The spinning conditions and dimensions of hollow pitch fibers obtained are listed in Table 2. The geometry of spinnerets used are shown in Figure 1.

As-spun mesophase and isotropic pitch fibers were stabilized at 320°C for 1 hour and at 270°C for 2 hours in oxygen, respectively. The carbonization for both fibers were carried out at 1000°C for 30 minutes in nitrogen.

The cross-sectional morphology of the hollow carbon fibers were observed with a field emission scanning electron microscope (JSM-6300F).

Results and Discussion

As seen in Figure 2 (a), hollow mesophase pitch-based carbon fibers were successfully prepared by spinning from pitch MP-1 at the temperature of 337°C using C-shaped spinneret. But, lower and higher spinning temperatures than 337°C for 5°C resulted in C-shaped and solid fibers [3]. When pitch MP-1 was spun through the triple-arc-shaped spinneret, it was found that the pitch streams leaving the spinneret can not combine with each other to form hollow fiber even at higher spinning temperature, resulting in separated arc-shaped pitch fibers. This phenomenon was attributed to the high softening point and high viscosity of pitch MP-1, which made the pitch melt exhibited very large viscous flow resistance and made the melt stream solidified too soon during the spinning.

However, the very low viscosity of the isotropic pitch at spinning temperature made it exhibited the tendency of slow solidification and hollow shrinkage under the action of surface tension. Therefore, the triple-arc-shaped spinneret, which has more and larger gaps between arc slits than the C-shaped one, had to be used to spin hollow fibers from pitch IP-1, even so, the resultant hollow fibers have rather small inner diameters, as seen in Figure 2 (b). Solid fibers were obtained from pitch IP-1, when C-shaped spinneret was used at any spinning temperatures.

References

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Acknowledgments

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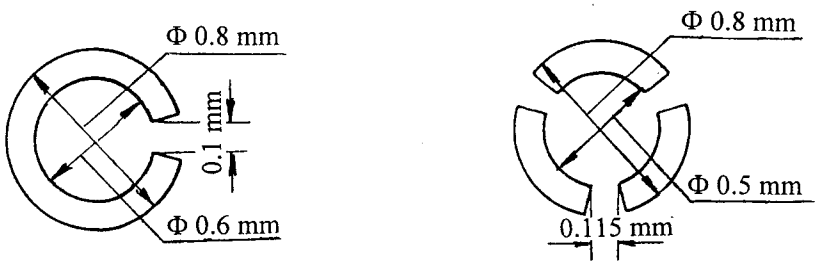
Table 1. Properties of precursor pitches

Pitch sample	Softening point (°C)	Anisotropic content (vol%)	Solubility (wt%)			Apparent viscosity* (Pa·s)
			BS	BI – QS	QI	
MP-1	295	100	4.7	24.1	71.2	84.1
IP-1	238	0	61.1	38.9	0	0.48

* Measured at spinning temperature shown in Table 2.

Table 2. Spinning conditions and dimensions of hollow pitch fibers

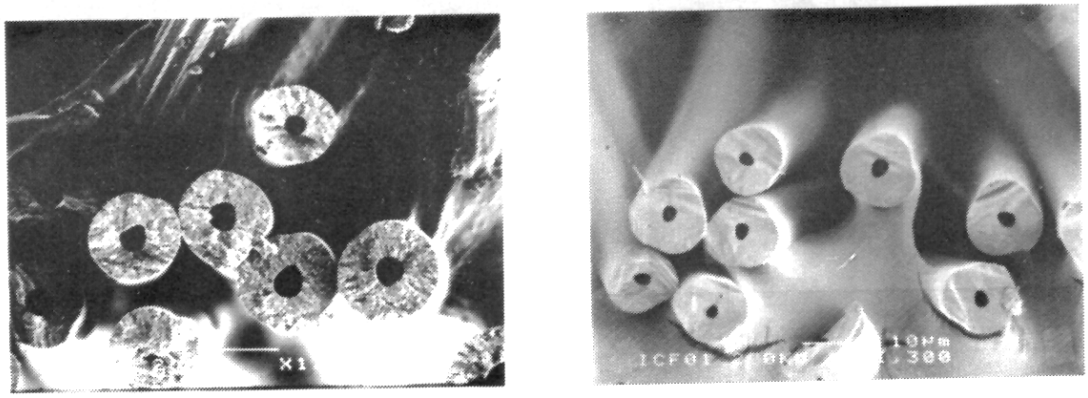
Precursor pitch	Shape of capillary	Spinning temperature (°C)	Spinning pressure (MPa)	Winding speed ($m \cdot min^{-1}$)	Outer diameter (μm)	Inner diameter (μm)
MP-1	C-shaped	337	0.6	340	24	7
IP-1	Triple-arc-shaped	265	0.5	620	16	3.5



Depth of spinneret = 0.60mm
(a) C-shaped

Depth of spinneret = 0.50mm
(b) Triple-arc-shaped

Figure 1. Geometry of spinnerets used.



(a) (b)

Figure 2. SEM photographs of hollow mesophase (a) and isotropic (b) pitch-based carbon fibers.