ICAM2009

11th International Conference
on Advanced Materials

Rio de Janeiro Brazil
September 20 - 25

Effects of stabilization time on microstructure of mesophase pitch-based carbon fibers with different diameters

G. F. Altoé(1)*, L. D. Castro(1) and L. B. Canto(2)

(1) NCDTC/ Centro Tecnológico do Exército, Rio de Janeiro, Brazil, e-mail: gisele.carbono@gmail.com
(2) UFRJ, Rio de Janeiro, Brazil
* Corresponding author.

Abstract – Mesophase pitch-based carbon fibers were produced from two pitches with different anisotropic contents (AC), stabilized in air using different residence times and carbonized under N_2 at 1000°C. The AC had high influence on the formation of the skin-core microstructure for the fibers treated with the lowest residence time. With the increase of the oxidation time, this effect was not observed and the results may indicate that the diffusion of the oxygen into the porosity of the fiber is the limiting factor for their complete stabilization.

High performance carbon fiber (HPCF) is the usual material employed in the construction of eolic devices used to generate electricity because it is strong and light. Pitch mesophase, a discotic nematic liquid crystalline material which has the capacity of forming graphite-like structure under heat treatment, is one of the precursors for HPCF. The heat treatment is carried out in three basic steps: stabilization, carbonization and graphitization. Stabilization involves the oxidation of the as-spun fibers to prevent them of melting during the carbonization step and also to keep the microstructure developed during the melt-spinning process. Completely stabilized fibers usually show a fine microstructure whereas partially stabilized fibers exhibit a skin-core structure with large domains of fusible material, which compounds the core [1].

Two petroleum-based mesophase pitches (Table 1) with different anisotropic contents were spun in a laboratory scale melt-spinning apparatus. Selected as-spun fibers (diameter of 15-65µm) were stabilized in air at 270°C for 30 and 300min and then, carbonized at 1000°C, under N_2 atmosphere. The microstructures of the cross-sections of the carbon fibers were observed under polarized light in optical microscope.

Pitch A fibers revealed skin-core microstructure for fibers with only 25µm of diameter, while Pitch B fibers presented this type of microstructure just for fibers with diameter larger than 35µm (Fig.1). For the same fiber diameter, the size of core formed in Pitch A fibers is larger than that formed in Pitch B fibers. Deformations were observed in fibers with diameter larger than 40µm for Pitch A and larger than 50µm for Pitch B probably because the thin skin could not contain the large molten core during carbonization. Fibers oxidized for 300min with diameter larger than 40µm showed skin-core microstructure for both pitches and no differences could be observed between the core sizes for Pitch A and B fibers, considering same range of diameter (Fig. 2). These results may indicate that for larger fibers diameter, the AC is not a determinant factor to reach some stabilization degree. In this case, oxygen diffusion into the porosity of the fiber may be the limiting reaction step. Fibers exhibiting skin-core structure showed onion-like layer arrangement or undefined arrangement with coarse domain. When the onion-like layer arrangement was observed it was noticed a tendency to produce a central hollow region during the carbonization process (Fig.3). It is not very clear the reason why this phenomenon took place and then further investigation is required.

Table 1: Properties of the petroleum pitches

Pitch	SP (°C)	AC (%)	QI (%)	TI (%)	ρ ($g.cm^{-3}$)	CY (%)
A	305	73	50.8	65.6	1.353	85
B	324	100	72.7	69.9	1.363	89

SP: softening point; AC: anisotropic content; QI: quinoline-insoluble; TI: toluene-insoluble; CY: coke yield.

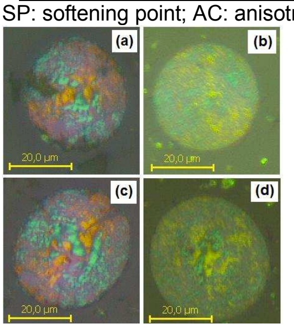

Figure 1: Micrographs of pitch A fibers (left) and pitch B fibers (right) oxidized for 30min. (a) and (b) show fibers with 32µm of diameter; (c) and (d) with 40µm.

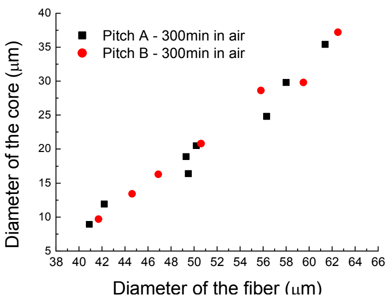

Figure 2: Diameter of core formed in the fibers as function of diameter for the fibers stabilized for 300min.

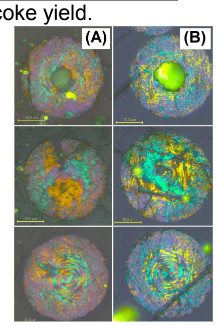

Figure 3: Micrographs of pitch A fibers (left) and pitch B fibers (right) showing different microstructures for fibers with the same range of diameter.

[1] S. Lu, C. Blanco, B. Rand. Carbon 40 (2002) 2109-2116.