Evolution of Structure and Properties in Continuous Carbon Fiber Formation

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A comprehensive study of the evolution of structure and properties in the formation of carbon fibers from acrylonitrile based precursors is being conducted in our laboratories. We report here some of the results regarding the effects of stabilization conditions on the formation of a consolidated carbon fiber structure. Results from a study of progression of carbonization in a continuous process are also described.

Experimental

Two precusor fibers were used in this study, one that was spun in our laboratories from a copolymer of acrylonitrile with 3% itaconic acid (precursor I) and other a commercial precursor. Different levels of orientational and lateral orders were obtained through various combinations of drawing through a hot water bath (HWD) and a high temperature tubular oven (HTD) (Table 1). Continuous oxidative stabilization was carried out in an 18-foot linear oven with various ascending temperature profiles (Table 2). The results reported here are from a continuous carbonization process with the temperature profile as shown in figure 1.

Table 1. Precursor I Drawing Conditions.

Sample	Draw Ratio		Total draw ratio	Final dpf
	B.W.	High Temp		
IA	3.0	-	3.0	2.25
IB	5.0	-	5.0	2.16
IC	2.5	1.8	4.5	2.22
ID	2.5	2.7	6.7	1.55

Table 2. Properties of Stabilized Precursor I Fibers.

SAMPLE	TEMPERATURE Profile(°C)	DPF	DENSITY gm/cc	HOLLOW CORE
IB	250-275-275	2.34	1.515	Yes
	250-275-300	2.29	1.525	No
	250-275-325	2.25	1.535	No
IC	250-275-300	2.19	1.495	Yes
	275-300-325	2.24	1.515	Rare
	275-300-350	2.24	1.530	No
ID	250-275-275	1.62	1.525	No
	250-275-300	1.57	1.535	No

Results and Discussion

The results from the SEM observations of cross sections of carbon fibers produced by continuous carbonization at 1 foot/min. of precursor I fibers stabilized with different temperature profiles are given in table 2.

A hollow core is seen from the carbon fibers when the fibers are incompletely stabilized as a result of lower stabilization temperatures, especially with the higher denier filaments. Extensive study with this precursor show that it is necessary to carry out the stabilization treatment until a critical density of 1.52 gm/cc. is reached in this precursor to avoid the formation of a hollow core in the carbon fibers. A hollow core develops in the carbon fibers also when the speed of the carbonization process is increased with apparently well stabilized fibers (Table 3). The formation of a hollow core can be caused by burning-off of an incompletely stabilized core as

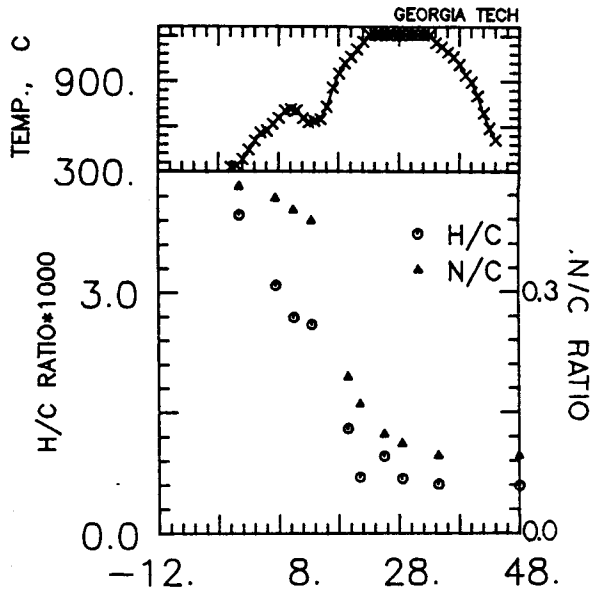

Figure 1. Elemental Composition and Progression of Carbonization.

Table 3. Carbonization at Different Speeds.

TAKE-UP SPEED ft/min.	RES. TIME AT 1200°C min.	HOLLOW CORE (SEM)	DENSITY gm/cc	SONIC MODULUS gpd
0.5	2.00	No	1.715	1061
1.0	1.00	No	1.705	1049
2.0	0.50	Yes	1.665	964
3.0	0.33	Yes	1.660	918

well as the rapid developement of a rigid skin and the subsequent developement of the carbon fiber structure inwards from the skin. These two aspects are being studied currently in our laboratories.

Higher orientational order as well as lower denier per filament in the precursor lead to superior mechanical properties of the carbon fibers (Table 4). However, the highest tensile strengths obtained from precursor I were still lower than the fibers from the commercial precursor II. The surface of carbon fibers made from precursor I displayed defects in the form of pits, presumably resulting from contamination during the formation of these precursor fibers.

Elemental composition and properties such as sonic modulus and electrical resistance change rapidly in the carbonization process, reaching their final values almost immediately upon reaching the highest temperature (figures1-3). The apparent density of carbon fibers also increases rapidly in carbonization but it reaches a relative maximum as soon as the maximum temperature is reached, with a significant drop upon continued heating at the highest temperature (figure 4). The drop in density which occurs without a change in sonic velocity and electrical resistance, is probably due to the conversion of some of the micropores from the accessible to inaccessible ones. This aspect will be studied with BET isotherms and mercury porosity measurements and the results will be presented at the conference, along with additional results from carbonization at 1600°C.

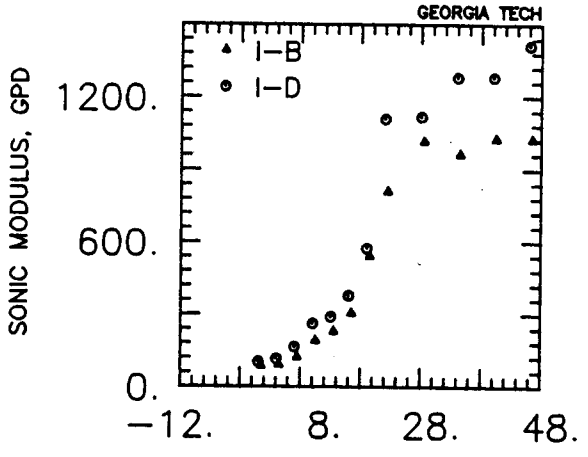

Figure 2. Sonic Modulus and Progression of Carbonization.

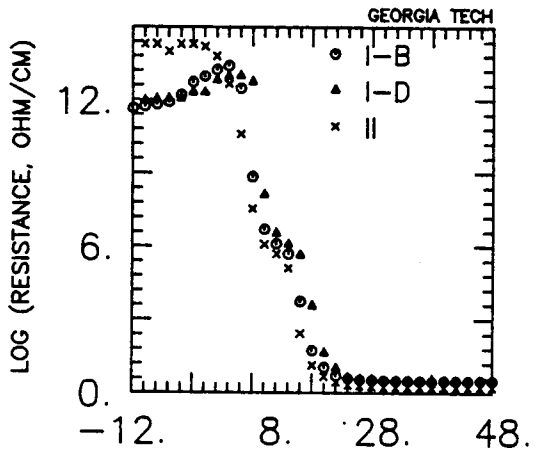

Figure 3. Electrical Resistance and Progression of Carbonization.

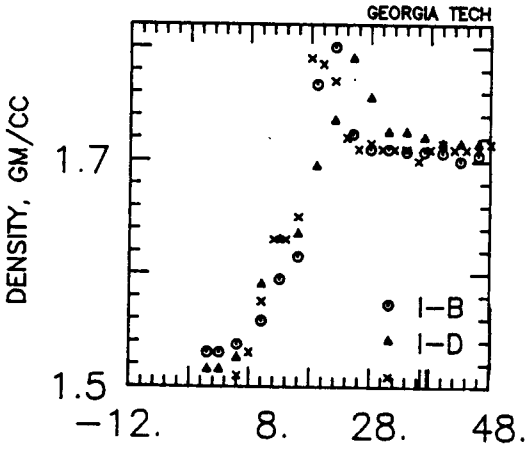

Figure 4. Density and Progression of Carbonization

Table 4. Properties of Precursors and Carbon Fibers.

SAMPLE	PRECURSOR FIBER					CARBON FIBER			
	DENSITY gm/cc	DPF	TENACITY gpd	ELASTIC MODULUS gpd	STABILIZATION TEMPERATURE (°C)	DENSITY gm/cc	DPF	TENACITY gpd	SONIC MODULUS gpd
IA	1.180	2.25	2.1	78	275-300-325	1.660	1.16	4.5	857
IB	1.175	2.16	3.1	90	250-275-300	1.705	1.13	6.3	1060
IC	1.175	2.22	3.4	114	275-300-350	1.670	1.10	7.4	1109
ID	1.185	1.55	4.4	144	250-275-275	1.715	0.83	8.1	1339
II	-	0.92	5.3	127	250-275-275	1.710	0.57	18.8	1210