## Thermotropic Polyester Carbonates. II. Polyester Carbonates as High Performance Fibers

#### Y. C. LAI,\* B. T. DEBONA, and D. C. PREVORSEK, Corporate Research and Development, Allied-Signal Incorporated, Morristown, New Jersey 07960

#### **Synopsis**

The fiber spinning and heat treatments of spun fibers from thermotropic polyester carbonates are described. Thermotropic polyester carbonates derived from t-butylhydroquinone, methylhydroquinone, diphenyl terephthalate, and diphenyl carbonate in the molar ratios of 50:50:55:45; 50:50:57.5:42.5; can be spun successfully, and after proper heat treatment, yield fibers with tenacity as high as 20 g/denier. Normally the best spinning temperature is  $10-20^{\circ}$ C above melting.

#### **INTRODUCTION**

Although the fiber spinning and the associated heat treatments of thermotropic polyesters have been reported in the patent literature,<sup>1</sup> there exists substantial gaps in the technology and scientific understanding of such systems and processes. In the thermotropic polyester systems disclosed in the patent literature fibers were spun in the thermotropic melt region giving tenacities and deniers which are typical for fibers such as Nylon-66. However, these thermotropic fibers can be heat-treated in the solid state either in nitrogen or in vacuum for times varying from several seconds to several hours or several days to reach tenacities as high as 15 to 20 g/d, qualifying them as high performance fibers.

In this paper, the fiber spinning and the heat treatments of spun fibers from thermotropic polyester carbonates will be described. All pertinent factors associated with spinning conditions and heat treatments of fibers were considered.

#### **EXPERIMENTAL**

#### **Polymer Samples Used for Study**

The samples used for study are those derived from *t*-butyl hydroquinone (tBuHQ), methylhydroquinone (MHQ), diphenyl terephthalate (DPT), and diphenyl carbonate (DPC) in the molar ratio of 50:50:50:50:50:50:50:50:55:42.5 and 50:50:60:40 and are listed in Table I. The preparations of those samples were described elsewhere.<sup>2,3</sup>

\*Present address: Professional Products R & D, Bausch & Lomb Inc., Rochester, NY 14692.

Journal of Applied Polymer Science, Vol. 36, 805-817 (1988)

© 1988 John Wiley & Sons, Inc.

CCC 0021-8995/88/040805-13\$04.00

ET ... O . to . to ... Oto ... it ...

Composition	Solution		
BuHQ/MHQ/DPT/DPC	in RV	mp (°C)	characteristics
50/50/55/45	1.79	250	good
	1.97	250	good
	2.78	250	good
	3.38	278	good
	6.95	294	poor
	12.7	299	poor
50/50/57.5/42.5	2.07	303	poor
	3.19	250	good
	6.63	270	good
	10.9	297	poor
	16.3	304	poor
	2.15	307	poor
50/50/60/40	2.97	289	poor
	3.10	289	poor
	6.70	299	poor
	9.59	299	poor
	insol.	308	poor
50/50/50/50	2.72	180	good

TABLE I

# Large-Scale Spinning-Using Brabender Extruder

The Brabender extruder was preheated to the desired temperature depending on the melting temperature of the sample being extruded. The dried powder sample was fed from the hopper. After melting, the melt was extruded through a 3-hole die and the fibers were either water quenched or air quenched and collected at various take-up speeds. Normally fibers of 100-500denier were obtained.

#### Small-Scale Spinning-Using Miniextruder

The extruder was heated to the desired temperature (normally  $250-316^{\circ}$ C), and the feeding zone was cooled with a water jacket to keep it in the  $140-160^{\circ}$ C range. Normally 3-hole dies with a radius of 0.02 in. were used; occasionally a 3-hole die with 0.013 in. or 1-hole die with 0.02 in. radius was also employed. After the desired temperature was reached, the screw speed was set to 52 rpm, the finely powdered (40 mesh) sample was then fed into the feeding zone and it advanced into the die area. When enough sample was fed into the barrel, the fibers started to extrude from the die and were taken up at a speed from 24 to 187 ft/min. The screw speed could also be adjusted to change the denier of fiber spun. Normally the die temperatures varied from the melting temperatures to 50°C above melting, and were increased at 10°C intervals. In most cases higher take-up speeds were preferred.

#### **Heat Treatment of Fibers**

If no load was desired, normally the two ends of fibers were bound to the two ends of 1-1.5-ft long glass tube with high-temperature tape. The fiber was

aligned diagonally along the inside of the tube. If some load was desired, the weights were placed on the middle of the fiber and the ends of fibers were tied to one end of the glass tube. This tube was mounted vertically. The heat treatments of fiber were then conducted in a Blue M oven (Model AGC170EMTI) or in a vacuum oven for the proper amount of time.

#### **Evaluation of Fibers**

The as-spun and heat-treated fibers were measured for any change in RV (reduced viscosity) during heat treatment. The solvent used for these measurements was p-chlorophenol/1,2-dichloroethane (50/50). RV results are expressed in dL/g.

The tenacity, modulus, and elongation of fibers were evaluated on an Instron testing machine using 1 in. gauge of fiber under controlled environment (23°C, 50% relative humidity, RH). Values for tenacity and modulus were obtained by manual calculations. Units were expressed in g/denier.

#### **RESULTS AND DISCUSSION**

#### Fiber Spinning Studies Using a Brabender Extruder and Heat-Treatments of the Resulting Fiber

A Brabender extruder was first chosen for spinning the thermotropic polyester carbonates, because it is used so routinely for screening. The general problems of using a Brabender extruder for thermotropic polymers are: (1) It consumes too much material in one operation. This made a systematic spinning study of limited quantities of thermotropic polyester carbonates difficult. (2) Because of its capacity and die characteristics, it makes drawing of fine denier fiber difficult without sample degradation. Because of these problems, only heavy denier fibers were obtained.

Normally, for samples with good fiber-forming characteristics, the color of yarn is good, except for samples with very high melting temperatures. However, the yarns are not uniform in cross-section.

Our heat-treatment studies on fibers started with many unknown factors such as temperature, time span, and conditions for heat treatment. For screening purposes, heat treatments of fibers were conducted by binding fibers in a glass tube with or without load at different temperatures for various periods of time either under nitrogen or in vacuum. Once the procedure for efficient heat treatment was established, only that procedure was applied. Every effort was made to handle the fiber carefully. The yarns become loosely bound in the glass after heat treatment, indicating the extension of the yarn. Sometimes the yarns were broken, partly due to excessive load on the yarn. Among the factors which affect the heat-treatment results are: solid-state polymerization kinetics, yarn surface/weight ratio, applied tension, and rate of polymer decomposition at the treatment temperature.

Because of the complexity of this process, analysis of the results is difficult. However, simple and meaningful conclusions still can be drawn.

Table II summarizes the results of spinning and heat treatments of spun fiber derived from tBuHQ/MHQ/DPT/DPC (50/50/50) with RV = 2.75. After spinning, the viscosity dropped from 2.72 to 1.89. Some conclusions

	Sample	Die tem	p. (°C)	RV	Denier	Tenacity g/d max./ave.	Modulus g/d max./ave.	UE % ave.
Coi	ntrol	22	8	1.89	584	4.0/3.5	183/150	3.6
					100	3.75/3.3	141/127	<b>4.1</b> <sup>·</sup>
Aft	er heat treati	ment in N <sub>2</sub> ove	en				,	
		Temp (°C)	Time (h)					
1	glass tube	210	6	4.76	71	6.3/5.1	163/130	5.2
2	glass tube	210	6		576	5.3/4.5	158/143	4.0
3	glass tube	215	6		110	7.2/5.3	164/122	5.7
4	glass tube	215	20	insol.	115	7.0/6.0	170/164	4.9
5	glass tube	215	20		45	8.2/7.1	182/167	6.2
	load 1.3 g						,	
6	glass tube	215	20		91	8.3/6.0	195/144	5.4
	load 2.6 g						,	
Aft	er heat treat	nent in vacuu	m oven					
7	glass tube	162	1		96	5.0/4.2	308/186	3.2
		172	1					
		186	1					
		208	1					
8	glass tube	200	12		112	5.0/4.1	206/157	3.8
9	glass tube	200	60		54	7.2/4.8	217/166	4.1
10	glass tube load 1.1 g	200	12		96	6.2/4.8	199/188	3.7

TABLE II Fiber Spinning and Heat Treatment of Thermotropic Polyester-Carbonate Derived from tBuHQ/MHQ/DPT/DPC (50/50/50), RV = 2.72

which can be drawn from these heat-treatment studies are:

1. Fine denier fibers can be heat treated better than more coarse ones; this is due to the larger surface/weight ratio, comparing samples 1 and 2. For the best cases, tenacity more than doubled (from 3.3 g/d to 7.1 g/d) for fine denier fiber. For the worst case, tenacity increased only slightly (from 3.5 g/d to 4.5 g/d) for the heavy denier fiber.

2. Heat treatments are more efficient at higher temperature. This is due to higher postpolymerization and annealing rates at higher temperature, comparing the samples 1 and 3.

3. As expected, longer heat treatment time yields better results in fiber properties (comparing example 1 and 4). However, the properties begin to level off after a prolonged time of heat treatment.

4. It is difficult to compare the results between heat treatments under  $N_2$  and under vacuum because of insufficient data. However, it is more convenient to conduct the process in an  $N_2$  oven than in a vacuum oven.

5. In general the fiber becomes thinner after heat treatment, particularly when a load is put on the fiber.

6. UE normally increases after heat treatment. However, modulus remains the same before and after heat treatment.

7. The molecular weight (RV) of heat-treated samples increases since postpolymerization occurs during the process.

This systematic study provided some guidelines for further studies on heat treatment of spun fiber.

	Sample	Die tem	p. (°C)	RV	Denier	Tenacity g/d max./ave.	Modulus g/d max./ave.	UE % ave.
C	ontrol	24	5	2.36	162	6.0/3.6	331/233	2.3
A	tter neat treat	ment in $N_2$ ov	en					
		Temp (°C)	Time (h)					
1	glass tube	235	15	insol.	165	7.8/5.5	280/176	3.4
	0	260	15			1		
2	glass tube	260	15	insol.	145	8.9/5.8	270/184	3.8
	load 3.9 gd							
3	glass tube	235	15					
		250	15	insol.	187	10.5/6.8	263/204	4.5
4	glass tube free end	250	15	insol.	165	9.4/6.6	263/199	4.4
5	glass tube load 3.9 g	250	15	insol.	169	9.3/5.9	285/203	4.5
6	glass tube	235	30	insol.	230	11.2/6.5	304/227	4.0
7	glass tube load 3.9 g	235	30	insol.	189	9.3/6.4	228/185	4.6

 TABLE III

 Fiber Spinning and Heat Treatment of Thermotropic Polyester-Carbonate Derived from tBuHQ/MHQ/DPT/DPC (50/55/55/45), RV = 2.78

Thermotropic polyester carbonate derived from tBuHQ/MHQ/DPT/DPC (50/50/55/45) RV = 2.78 was spun with a die temperature of 245°C. The heat treatments of fibers were conducted in an N<sub>2</sub> oven only, since we found it was more convenient and efficient. Table III lists the heat-treatment data for the as-spun fibers. It is interesting to note that even though the fibers were very thick, the tenacity still increased by 50 to 90% after proper treatment and not much drop in RV (from 2.78 to 2.36) occurred during spinning. The conclusions drawn above still hold true. Fibers of polyester carbonate derived from tBuHQ/MHQ/DPT/DPC (50/50/60/40) with RV value of 2.15 were also obtained (see Table IV). Because of the high processing temperature, considerable decomposition (RV = 1.46 after extrusion) occurred and the as-spun fiber showed a disappointing tenacity of 2.1 g/d. For this material heat treatments gave the best result when lower temperatures were used.

From the study of fiber spinning by using a Brabender extruder and heat treatments of fibers in an  $N_2$  oven, it is evident that the polymers can be spun without major difficulty, but generally only high denier as-spun fibers can be obtained. The as-spun fiber can be heat treated to give up to 100% improvement in tenacity. However, in order to maximize the fiber properties of existing compositions, some improvement in the design of the extruder, particularly for obtaining fine denier fibers, was necessary.

#### Use of Miniextruder for Fiber Spinning

A miniextruder constructed with good characteristics in heating, cooling, flow, and extruding was used for further systematic fiber-spinning studies of thermotropic polyester carbonates.

	Sample	Die tem	p. (°C)	RV	Denier	Tenacity g/d max./ave.	Modulus g/d max./ave.	UE % ave.
C	ontrol	29	5	1.46	57	2.6/2.1	258/170	1.2
A	fter heat trea	tment in N <sub>2</sub> ov	/en					
		Temp (°C)	Time (h)					
1	glass tube	280	18	insol.	105	5.7/4.8	217/177	3.6
2	glass tube	280	18	insol.	71	4.9/3.5	278/199	2.4
	load 1.3 g							
3	glass tube	280	18	insol.	74	7.0/3.8	283/233	2.1
	load 2.6 g							
4	glass tube	280	18	insol.	75	4.0/2.5	209/143	2.1
	load 3.9 g							
5	glass tube	280	30	insol.	69	3.2/2.1	277/188	1.4
6	glass tube	280	30	insol.	61	4.2/2.6	247/198	1.7
	load 1.3 g							
7	glass tube	280	30	insol.	56	3.6/2.7	312/248	1.4
	load 2.6 g							

TABLE IVFiber Spinning and Heat Treatment of Thermotropic Polyester Carbonate Derivedfrom tBuHQ/MHQ/DPT/DPC (50/50/60/40), RV = 2.15

Most samples listed in Table I were spun from this miniextruder. Most of these samples were obtained from heat treatments of polymer powders as described elsewhere.<sup>2</sup> In general, materials with RV < 3.5 and melting temperatures below 290°C can be drawn into fine denier fibers under proper spinning conditions, and less degradation occurred during the process. However, with materials of higher RV's and thus high melting temperatures, severe degradation of the sample occurred in the barrel such that the fibers could not be drawn properly. In cases of the most severe decomposition, no fibers could be formed. Because of less degradation for low RV samples, more systematic studies such as using different die hole sizes, 3-hole or 1-hole dies, and drawing at different temperatures, could be carried out. But for samples with high RV, fibers could be drawn only at the lower temperatures, and no work could be done at higher temperatures because of severe degradation.

The following describe the results of fiber spinning using the miniextruder and associated heat treatments.

Thermotropic polyester carbonate derived from tBuHQ/MHQ/ DPT/DPC (50/50/55/45). Polymer of this composition was the one studied most extensively because at high RV it has a lower melting temperature compared to those polymers containing higher carboxylate ester content. Table V lists the fiber spinning and heat-treatment studies on this composition. The study of this series was started with the sample of lowest RV (1.97), spun at the lowest possible temperature and fibers were taken-up at medium to high speed. Then higher extrusion temperatures were used, followed by spinning of samples with higher RV's. Because of the large number of factors under study, the measurements of tensile properties were made selectively on those fibers which appeared to offer the most promising results. Those at

Fiber Spinning and Heat Treatment of Thermotropic Polymer Derived from tBuHQ/MHQ/DPT/DPC (50/50/55/45) TABLE V

				As-spur	ı fiber			Hea	t treated at 240°	C, 6 h N <sub>2</sub>	:
RV before/after extrusion	$T_m$ (°C)	Die temp. (°C)	Denier	Tenacity g/d max./ave.	Modulus g/d max./ave.	UE % ave.	RV	Denier	Tenacity g/d max./ave.	Modulus g/d max./ave.	UE % ave.
1.97/1.90	250	252	60	5.5/2.3	266/205	2.9	insol.	52	7.7/4.6	289/202	4.5
			41	4.8/3.1	325/219	1.5	insol.	37	9.1/4.8	335/287	3.9
		263	30	3.9/3.1	346/199	2.1	insol.	20	14.0/10.3	341/243	4.1
		266	19	6.6/5.0	343/280	2.2	insol.	13	20.0/13.4	357/280	4.9
1.97/1.67	250	264	38	3.8/2.5	210/150	2.0	insol.	19	10.0/6.9		3.3
1.97/1.70	250	271	50	2.9/2.0	180/151	1.4	insol.	38	9.6/7.4		4.0
		282	39	3.7/3.0	198/188	2.0	insol.	17	8.6/6.4		3.8
		282	26	3.7/2.5	184/157	1.6	insol.	17	8.7/6.8		4.1
3.38/2.23	278	330		too brittle							
6.95/2.74	294	330		too brittle							
9.45	299	330		no fiber forming							
12.7	303	330		no fiber forming							

## THERMOTROPIC POLYESTER CARBONATES. II

lower take-up speed and/or with weak fiber appearance were excluded from measurements.

This systematic study provided the following information:

- a. Samples with low RV (1.97) experienced little or no drop in viscosity after extrusion at temperatures only moderately higher than melting. (In fact, drop in RV occurred only when very high extrusion temperature was applied). However, samples with high RV encountered serious drop in RV after extrusion, because of decomposition at the required high barrel temperatures. (For example, after extrusion, the sample with RV of 6.95 can drop to 2.74 at 330°C.)
- b. Regardless of the starting RV of the spun fibers, all fibers can be heattreated at 240°C for 6 h under  $N_2$  oven to give samples which are insoluble in the same solvent system used for measuring the viscosity of as-spun fiber or initial polymer. This indicates that thermotropic polyester carbonates can be heat-treated much more efficiently when they are in fine denier fiber form (50 denier or lower). For comparison, the sample of similar viscosity (1.97) treated in powder form (40 mesh) under  $N_2$  oven at 240°C can be treated to RV 4.19 after 6 h; 6.95 after 12 h and 12.7 after 24 h.<sup>2</sup> Apparently, high surface area/weight ratio plays an important role here.
- c. Samples with lower RV's give fibers which can be drawn with higher take-up speed and thus yield finer denier fibers which, after heat treatment, yield higher tenacity fiber. On the contrary, fibers spun from high RV material are usually very weak and cannot be drawn at higher take-up speed. This is due to the difference in melting temperature and the high quenching rate in air. (Using a heated sleeve under the spinneret helps the drawing of higher melting material.)
- d. Samples of low RV (1.97) may not necessarily give the best tenacity in terms of the as-spun fibers, however, if drawn at an appropriate temperature 10 to 20°C above melting, with high take-up speed, followed by appropriate heat treatment (N<sub>2</sub>, 240°C, 6 h), tenacities as high as 20 g/d can be reached (average 13 g/d).
- e. Heat-treatment does not appear to affect the tensile modulus of the fiber. However, UE values increase after heat treatment. Typical increases from 1.5-2.9 to 3.3-5.0% are observed.
- f. Heat treatment for a longer time does not necessarily give better fiber properties.
- g. Good fiber properties (after heat treatment) can be obtained only when the fiber has a low denier (20 denier or less).
- h. Compared with the heat treatments of thermotropic polyester fibers cited in the patent literature, our heat treatment procedure is a very efficient one. To obtain fiber with the same level of tenacity, most thermotropic polyester fibers need higher heat-treatment temperature and/or longer heat-treatment time. The reason for this is most thermotropic polyesters were prepared from carboxylic acids and acetates of aromatic hydroxy compounds and a higher temperature is required for transesterification to take place, compared to the reaction of phenyl esters and aromatic diols used with the polyester carbonates.

Fit	oer Spinnin	g and Heat-Treat	ment of Th	TA lermotropic Polyı	BLE VI mer Derived fror	n tBuHQ	/MHQ/	DPT/DP	C (50/50/57.5/42	2.5)	
				As-spur	ı fiber			Hea	t-treated at 240°	C, 6 h, N <sub>2</sub>	
RV before/after extrusion	$T_m$ (°C)	Die size & temp. (°C)	Denier	Tenacity g/d max./ave.	Modulus g/d max./ave.	UE % ave.	RV	Denier	Tenacity g/d max./ave.	Modulus g/d max./ave.	UE % ave.
3.19/2.24	288	3-hole 0.02 in									
		299	40	5.1/3.7	315/237	2.1	insol.	44	9.2/8.4	354/282	3.7
			22	5.3/3.8	317/254	1.9	insol.	11	14.5/11.0	384/297	3.8
			15	5.6/4.0	324/241	2.0	insol.	12	18.1/13.6	367 /274	4.1
2.07/1.85	250	<b>3-hole 0.02 in</b>	6	5.4/4.1	284/223	2.1	7.16	18	11.1/7.9	303/255	3.8
		260	7	8.9/4.8	402/279	24.1	7.16	9	18.2/16.5	410/285	3.9
2.07/1.79	250	1-hole 0.02 in									
		260	20	5.5/4.1	387/243	1.9	insol.	20	8.0/6.5		
			20	7.1/3.8	410/249	1.8	insol.	13	12.9/8.8		
		263	20	4.1/3.3	331/235	1.7	insol.	6	14.2/9.8		
2.07/1.65	250	<b>3-hole 0.013 in</b>									
·		260	24	4.0/2.0	281/179	1.1	insol.	24	9.1/7.1		
			12	3.8/3.2	209/178	2.0	insol.	14	10.6/8.7		
		274	12	5.5/3.0	311/228	1.5	insol.	6	6.6/4.6		
		288	17	3.2/2.4	405/231	1.2	insol.	15	7.6/4.7		
		304		Difficult to	o take-up						

THERMOTROPIC POLYESTER CARBONATES. II

		UE % ave.			
for Extrusion	3, 5 h, N <sub>2</sub>	Modulus g/d max./ave.			
ing Miniextruder	-treated at 240°C	Tenacity g/d max./ave.	8.3/6.0 6.7/3.8	4.5/2.4	
/40), by Us	Heat	Denier	11 14	7	
(20/20/60		RV	insol. insol.	insol.	
T/DPC		UE % ave.	$1.9 \\ 2.1$	1.2	
TABLE VII buHQ/MHQ/DF	fiber	Modulus g/d max./ave.	369/226 434/328	368/297	
tropic Polymer tF	As-spun	Tenacity g/d max./ave.	6.5/3.3 7.6/4.8	3.1/2.9	
of Thermo		Denier	10 17	10	
eat-Treatment		Die temp. (°C)	299		
g and He		т <sup>"</sup>	289		
Fiber Spinnin		RV before/after extrusion	2.97/1.48		

Thermotropic polyester-carbonate derived from tBuHQ / MHQ / DPT / DPC (50 / 50 / 57.5 / 42.5). Samples of lower RV's (3.19 and 2.07) of this composition were studied in a manner similar to their low RV counterparts of the previous composition. Samples of high RV were also screened, but results were generally poor.

Table VI lists the data on fiber spinning and heat-treatment studies for this composition. As expected, finer denier fibers from high take-up drawing gave the best results in terms of tenacity after heat treatment. No serious drop in RV was associated with extrusion of low RV samples. Conclusions cited in previous sections still hold true here. In addition, the following important observations are noted:

- a. The spun fibers with original RV = 3.19 (melting temperature 288°C) drawn at 299°C were taken up at three different speeds. Those parts of the fiber with the highest take-up speed had the highest tenacity 18.1 g/d (average 13.6 g/d) after heat treatment; those parts with the lowest take-up speed had the lowest tenacity 9.2 g/d (average 8.4 g/d) after treatment. Those with medium take-up speed had an intermediate tenacity.
- b. Finest denier (7) fibers were obtained from the sample with RV = 2.07 spun at 260° with highest achievable take-up speed (374 f/m). The fibers had a tenacity of 18.2 g/d (average 16.5 g/d) after heat treatment.
- c. For the same sample, as expected, higher processing temperatures gave poorer results. One reason for this is the degradation of the sample when the processing temperature is too high. Another reason may be the decrease in liquid crystalline orientation at higher die temperatures.

Theoretically, thermotropicity (or liquid crystallinity) increases with temperature once beyond the melting point. But from the experience accumulated from the fiber-spinning studies (and separate injection molding studies), it was found that if the material is processed at temperatures  $10-20^{\circ}$ C above the melting temperature, it would show the best fiber properties (e.g., tenacity); and if the material was processed at temperatures  $30^{\circ}$ C above melting, the properties drop as processing temperature increases. The most likely reason for this is the decrease of thermotropicity for these particular compositions provided that degradation is not too severe at that temperature.

Thermotropic polyester carbonate tBuHQ / MHQ / DPT / DPC(50 / 50 / 60 / 40). Table VII lists the data for spinning and heat treatment of fibers derived from this composition. For obvious reasons no work was done on high viscosity samples. Even though the low RV (2.97) sample was drawn into fine denier fiber, the tenacities of the heat-treated samples were very low.

#### SUMMARY

From the results of fiber spinning and heat treatment of spun fibers conducted on samples derived from tBuHQ/MHQ/DPT/DPC by using either

	Summary of th	ne Best-Fe	ertorming Fibers Spu	5			
					After heat tr	eatment	
Composition tBuHQ/MHQ/DPT/DPC	RV origin/as-spun/after heat-treatment	$T_m$	Spinning temp. die size	Denier	Tenacity g/d max./ave.	Modulus g/d max./ave.	UE % ave.
50/50.57.5/42.5	1.97/1.90/insol.	250	266°/3 hole 0.02 in	13	20.0/13.4	357/280	4.9
			263/3 hole 0.02 in	20	14.0/10.3	341/243	4.2
50/50/57.5/42.5	2.07/1.85/7.16	250	260/3 hole 0.02 in	9	18.2/16.5	410/285	3.9
			260/1 hole 0.02	13	12.9/8.8		
			263/1 hole 0.02	6	14.2/9.9		
50/50/57.5/42.5	3.19/2.24/insol.	280	299/3 hole 0.02	11	14.5/11.0	384/297	3.9
				12	18.1/13.6	367 / 274	4.1

TABLE VIII mary of the Best-Performing Fibers (

816

### LAI, DEBONA, AND PREVORSEK

#### a Brabender extruder or the miniextruder, it is clear that:

- 1. Thermotropic polyester carbonates can be spun to fine denier fibers, which upon proper heat treatments can give a fiber with an average tenacity of 16.5 g/d. We believe that if the processing conditions are optimized, even better tenacity can be achieved, approaching or exceeding that of some thermotropic polyesters.
- 2. Thermotropic polyester carbonates of the composition described with melting temperatures in the range of 220-280°C and of suitable reduced viscosity (1.7 to 3.5) can be drawn under appropriate conditions into fine denier (7) fiber which has tenacity as high as 20 g/d after appropriate heat treatment.
- 3. The heat-treatment process developed in this study is very efficient in terms of the heat-treatment temperature/time applied.
- 4. Compared with some thermotropic polyesters reported,<sup>4</sup> polyester carbonates give lower tensile modulus. This is probably because the presence of carbonate groups reduces the degree of liquid crystallinity.

It was our intention to balance properties against process temperature. Our objective was to get a lower process temperature with carbonates but approach the properties of wholly aromatic polyesters. That is, polyester carbonates can be processed at similar conditions as conventional fibers (nylon, PET) and give much better tenacity.

Table VIII summarizes the best fiber spinning and heat-treatment results. Although satisfactory results have been obtained with fiber spinning/heat treatments, some problems remain unsolved, like how to carry out large-scale spinning to obtain fine denier (< 20 denier) fibers and how to heat-treat fibers in a continuous fashion. It is our belief that if more development work is conducted these problems will be solved.

#### References

- 1. R. R. Luise, U.S. Pat. 4,183,895 (1980).
- 2. M. K. Akkapeddi, B. T. DeBona, Y. C. Lai, and D. C. Prevorsek, U.S. Pat. 4,398,018 (1983).
- 3. Y. C. Lai, B. T. DeBona, and D. C. Prevorsek, Macromolecules, submitted for publication.
- 4. U.S. Pat. 4,083,829, 4,256,62 (Celanese), and 4,159,365 (DuPont).

Received July 16, 1986 Accepted September 30, 1986