Manmade Fibers: Their Origin and Development

Edited by

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ELSEVIER APPLIED SCIENCE

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Dr Raymond B. Seymour, Prominent Polymer Scientist and Educator

Raymond B. Seymour, an inventor and researcher who dedicated much of his working life to polymer education, died on November 15, 1991. He was 79. Seymour died at his home in Hattiesburg. He had been Distinguished Professor of Polymer Science at the University of Southern Mississippi.

Ray Seymour had earned both a bachelor's and a master's degree in chemical engineering from the University of New Hampshire. In 1937 he earned a doctorate from the University of Iowa.

In 1989 the Society of Plastics Engineers presented Dr Seymour with its highest award, the International Gold Medal. During his career, Seymour received more than 40 other professional honors and awards, wrote more than 1500 scientific reports and helped develop products protected by 45 US patents and 100 patents in foreign countries.

He demonstrated plastics on television science shows. He wrote books ranging from technical works to an introductory text for grade school children. In the 1940s, Dr Seymour introduced the first undergraduate course in polymer science.

FOREWORD

Since the recording of the history of various phases of polymer science is of the utmost importance, it was reassuring to note the previous publications on the history of polymer science, edited by Professor Seymour and published by Marcel Dekker, Inc. in 1981; *Polymers: Their Origin and Growth as a Science*, authored by Dr H. Morawetz and published by Wiley in 1985; *Origin and Development of High Performance Polymers*, coedited by Drs Seymour and G. Kirschenbaum and published by Elsevier Science Publishing Co. in 1986; *History of Polyolefins*, coedited by Drs Seymour and T. Cheng and published by Reidel in 1986; and *Pioneers in Polymer Science*, coedited by Drs Seymour, H. Mark, L. Pauling and C. Marvel and published by Kluwer Academic in 1989.

It was also my pleasure to serve as coeditor of *Organic Coatings: Their Origin and Development*, which was published by Elsevier Science Publishing Co. in 1990. Fortunately, Drs Seymour and Porter have added to the history of polymer science literature by coediting this book on *Manmade Fibers: Their Origin and Development*. As was the case with other books in this series, many of the chapters are written by pioneers in polymer science who were responsible, to a large extent, for today's polymer industry.

H. MARK
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PREFACE

Natural fibers, such as wool, silk and cotton, have been used for many thousands of years. However, the possibility that manmade fibers could be synthesized was overlooked until three centuries ago when Hooke in his *Micrographia* suggested that man could utilize artificial substances that could be wire drawn, like the silkworm's 'clew'. De Réaumur promoted Hooke's suggestion in 1774, but a useful filament was not produced by man until Swann and de Chardonnet extruded a solution of cellulose nitrate (collodion) through small holes (spinnerets) in the last part of the 19th century.

These pioneer manmade fibers were replaced by rayon fibers, which were spun from an alkaline cellulose xanthate solution (viscose) and were subsequently supplemented by cellulose acetate and many synthetic fibers, such as nylon, polyesters, polyolefins, and acrylic fibers.

Much has been written about the history of natural fibers but very little has been written about the history of manmade fibers. Chapters on the history of natural and synthetic fibers by Drs Charles Fisher and Paul Morgan, respectively, were published in *History of Polymer Science and Technology*, which was published by Marcel Dekker, Inc. in 1982.

According to Fisher, about 15 million tons of natural fibers are produced annually. Their history is not discussed in this book, but considerable information is presented on the history of manmade fibers which according to Morgan are produced at an annual rate of about 36 million tons.

RAYMOND B. SEYMOUR
ROGER S. PORTER

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The editors would like to thank the many authors who presented reports at the International Symposium on History of Manmade Fibers at the National Meeting of the American Chemical Society at Atlanta, GA on April 17–19, 1991. The editors also appreciate the assistance of Ms Paula E. Phillips and Mrs Mischa Thomas, who aided in the preparation of this book.

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AN UPDATE ON FIBER SCIENCE AND TECHNOLOGY

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The original fibers were natural products, such as flax and cotton, which were obtained from plants and animal products, such as wool and silk. The advance of textile arts, such as spinning and weaving, paralleled other advances in civilization from the Stone Age to the Industrial Revolution and to the present era. Mechanization from the cotton gin to the spinning jenny catalyzed a new era in the textile industry and made cotton king.

Manmade fibers, such as regenerated cellulose (rayon) and cellulose acetate (acetate rayon), which were commercialized in the early 1900's, provided a new source of fibers, but cotton continued to be the principal fiber until after the advent of the synthetic fibers, viz, nylon, polyester and acrylic. Cotton accounted for less than 50 percent of the 3.8 million tons of fibers used in the U.S. in 1965, and this share continues to decrease. Twenty-five kg per capita of fibers were consumed in the U.S. in 1925 and manmade and synthetic fibers accounted for 16kg per capita or 69 percent.

The worldwide production of fibers in 1975 was 23.7 million tons and the product mix, in millions of tons, was as follows: synthetic fibers (7.4), rayon and acetate (3.0), cotton (11.9), wool (1.5), and silk (0.02). The growth of the fiber industry continues at a rate that is 50 percent greater than that of the population growth [1]. The production of synthetic fibers, worldwide, increased by about 6 percent in 1987 [2].

Western Europe accounted for 38 percent of the world's output of synthetic fibers in 1987, Japan accounted for 16 percent, and the U.S. accounted for 11 percent. Seven and one-half million tons of polyester fibers were produced, worldwide, in 1987, and the output by various countries, in percent, was as follows: China (9%), Japan (7%), other Asian nations (30%), U.S. (12%), other American nations (7%), Western Europe (13%), Eastern Europe (10%), and Middle East, Africa, and Oceania (2%). Nylon, which was the number 2 synthetic fiber, was produced at an annual rate of 4 million tons, and the worldwide production of acrylic fibers in 1987 was 3.5 million tons.

The production of noncellulosic and cellulosic manmade fibers, in thousands of tons, in European nations in 1987 was as follows:

Country	Noncellulosic	Cellulosic
West Germany	821	16
Italy	655	3
Spain	268	0.5
Turkey	260	1
U.K.	227	12
Benelux countries	176	5
France	173	2
Switzerland	95	0
Ireland	78	0

Country	Noncellulosic	Cellulosic
Portugal	63	0.1
Austria	27	120
Finland	1	54

The distribution between filament and staple synthetic fibers, worldwide, is 57 percent and 43 percent, respectively [3]. The production of synthetic fibers in the U.S. and the entire world, in thousands of tons, in 1988 was as follows:

Fiber	U.S.	World
Polyester	1,682	7,718
Nylon	1,261	3,718
Polyolefin, vinyl	784	
Acrylic	275	2,523
Total	**3,413**	**13,959**

The production of rayon and acetate rayon, in thousands of tons, in the U.S. in 1988 was 1.8 and 0.95, respectively. Autex closed its rayon plant at Front Royal, VA, but reopened after receiving a long-term contract from NASA [4,5]. Forty tons of aerospace grade rayon is required for lining a pair of shuttle booster nozzles in order to hold the temperature of the nozzle below 100°C. The lining is carbonized and coated with a phenolic resin coating before firing. Other U.S. producers of rayon are BASF, Courtaulds, Enka, and North American Rayon.

The production of fiberglass, in thousands in tons, in the U.S. in 1988 was as follows:

Owens Corning	182
PPG	155
Certain Teed	57
Manville, etc.	11
Total	**405**

Since Amoco was the only domestic source of carbon fibers and most of these fibers were imported from Japan, the U.S. Government requested that at least 50 percent of the domestic requirement of carbon fibers be produced in the U.S. [6]. The annual worldwide consumption of carbon fiber is 2.7 thousand tons and 2 thousand of this fiber is consumed in the U.S. The U.S. aerospace industry consumed one thousand tons of carbon fiber in 1988.

As shown in the following table, there will be an annual capacity for the production of 3.8 thousand tons of carbon fiber by U.S. firms in 1989.

Hercules	1.2	Zoltech	0.11
Amoco	0.9	Polycarbon	0.45
BASF (Celion)	0.45	Textron	0.45
Courtaulds	0.41	Witco	0.23
Ashland	0.14		

Most of this fiber will be wet spun from acrylic fibers but Zoltech will use rayon as the precursor. Ashland will use pitch and 25 percent of Amoco's production will be from pitch at Greenville, SC. Celion will produce its carbon fiber by melt spinning [6] at Rock Hill, SC, and Williamsburg, VA [7]. Courtaulds will produce its carbon fiber in a joint venture with Dexter. Amoco will produce its fiber at Sacramento, CA, and Ridgefield, CT, and Hercules will produce carbon fiber at Magna, UT, and at Decatur, AL, in a joint venture with Sumitano [8].

Enimont produces polyester, nylon-66, and rayon, and is attempting to solve Italy's troubled fiber industry. The polyester facilities at Fayetteville, NC, and Darlington, SC, which Hoesch was forced to sell after its acquisition of Celanese, were sold to Fiber Industries which also acquired the tradename Fortrel [9]. Fiber Industries' sales in 1988 exceeded $350 million. Rhone Poulenc is investing $46 million to upgrade its polyester and nylon production facilities in several European nations.

Mann has acquired the acrylic fiber business at Williamsburg, VA, from BASF. Allied Signal is expanding its fiber production facilities at Moncure, NC, to 63 thousand tons. Hoechst is also expanding its fiber facilities at Bodingen, West Germany, and Sao Paulo, Brazil [10]. Monsanto has sold its Astroturf business to Balsam of West Germany for $15 million. Eastman has phased out its 110 thousand ton polyester business at Columbia, SC, but has retained its 73 thousand ton facility at Kingsport, TN. Alavere is spending $240 million to expand its polyester staple fiber capacity by 50 thousand tons at Shelby, NC, but has phased out its polyester yarn facility at Spartenburg, SC.

The mechanical properties of wood fibers have been improved by grafting with acrylic or ethylene monomers [11]. In a joint effort, Arco and DuPont are attempting to improve the water absorbency of textiles for the disposable diaper market which consumed 90 thousand tons of material in 1984 [12]. Formaldehyde-free wrinkle-free cotton fabrics are being made by crosslinking cellulose with 1,2,3,4-butane tetracarboxylic acid [13].

Investigations at the University of Tokyo have improved the properties of PET by swelling the imperfect crystals in acetone and drawing to produce more perfect crystals [14]. Monsanto has improved its synthetic fiber operations at Pensacola, FL, by balancing its energy requirements, etc. [15]. Monsanto is also producing nylon filament carpet with anion sulfonate surface groups [16].

Because of a decrease in demand for knitted textiles, the sale of acrylic fibers has decreased. Toray is promoting the use of acrylic staple fibers as replacement for asbestos fibers in Portland cement composite sheets. In addition to improving the quality of its acrylic fibers in the U.S., DuPont is aiding comparable improvements at Celbras Qinica at Aratu, SA [17]. Facilities for an annual production of 120 thousand tons of polypropylene (PP) fiber are being built at Dunkirk, using Union Carbide technology [18]. Amoco is expanding its annual PP yarn production by 10 thousand tons at Bainbridge, GA. Exxon, which is the leading supplier of spun bonded fabrics is using Reicofil technology in which PP filaments are kept separate until they are bonded in the mat [19].

Exxon has donated its melt flow non-woven fiber unit to the University of Tennessee at Knoxville. DuPont, which is the leader in nylon carpet face yarns, has purchased Hercules $50 million carpet fiber business and is now promoting PP face yarn under the tradename of "Pro Select" [20].

Exxon has licensed the heat bonded, non-woven "Teram" PP process from ICI.

DuPont is investing $150 million in its Tyvex spun bonded HDPE facility at Luxembourg. Geotextiles, which are superior to clay for stabilizing soil, are being used for road building and for increasing the capacity of landfills [21]. HDPE grid materials (Geogrid) are being used in several projects to control flooding [22]. The use of various polymers for geotextiles has been reviewed [23].

The use of vinyl-coated textiles is increasing. The name Ebb Tide is now being used instead of Naugahide for coated fabrics. The use of acrylic and nylon fiber-flocked textiles is also increasing [24]. The relationship of strength and length of carbon fibers has been investigated [25]. Melt-spun carbon fibers have non-circular cross sections. A new book on carbon fibers is available [26]. Fiber Materials (FM) is producing metal-filled and metal-coated quartz fibers at Columbus, OH [27].

Polybenzamide fibers (H-Cel), which are being added to aluminum to decrease cracking tendencies, are being considered as substitutes for asbestos for brake linings. Spider silk, consisting of crystalline and amorphous regions, which is being produced by bioengineering, is being considered for dragline silk [28]. Rovings are being admixed with resins and pultruded to provide high performance composites [29].

The court in U.K. upheld Akzo's patented infringement case on Twaron vs. DuPont's Kevlar. The two firms have agreed to cross license but Akzo is prohibited from selling its *p*-phenylene terephthalamide in the U.S. [30]. Tejin continues to produce this filament under a process that does not conflict with DuPont's patent claims. The present market for these aramid fibers is 10 thousand tons in the U.S., 5 thousand tons in Europe, and 1 thousand tons in Japan. A new aramid, Kevlar 149, which is stiffer and more moisture resistant than conventional Kevlar, is being used as a reinforcing fiber for composites in helicopter blades. GE has developed a hollow liquid crystal polymer (LCP) filament, which provides composites analogous to wood.

Allied Signal continues to produce extended chain HDPE (Spectra) using a DSM process but has discontinued R & D efforts on LCP filaments [31]. Fiberglass continues to account for 93 percent of reinforcing fibers but carbon, aramid, polyester, boron nitride, and phosphate (Fiberfax) fibers are also being used in resin composites. Continuous filaments with low dielectric constants, are being produced from boron, boron carbide, silicon nitride, and silicon carbide. Silica, alumina, metal oxide fibers, and whiskers also being used for the production of composites [32].

The properties of ceramic fiber-polymer composites with alumina (Fiber FP), alumina/silica (Nextil, Sumica), silica/silicon carbide/carbon (Nicalon) and amorphous silica have compared with those made from E glass, carbon, aramid, and quartz [33]. The annual production, in thousands of tons, of high performance fibers in 1987 and for 1992 are shown below [34]:

Fiber	**1987**	**1992 (est)**
Carbon	2.0	4.3
Aramid	10.0	16.0
S Glass	2.5	4.4
Ceramics	0.16	0.7
Boron	0.16	0.2
Quartz	0.16	0.2
HDPE (Spectra)	0.4	1.0
Total	**14.5**	**26.0**

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NEW HORIZONS IN THE FIBER INDUSTRY

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Introduction

The growth and harvesting of natural fibers and the production of manmade fibers is one of man's most important activities, worldwide. Nature has been particularly generous in supplying cellulose fibers, such as cotton (Gossypium hirsutium), linen (Linum usitatissimum), hemp (Cannabis sativa), jute (Boehmeria nivea), sisal (Agave sisallina), kapok (Ceiba pentandra), and coir (Cocos Nocitera). Proteinaceous fibers such as wool, mohair, cashmere, alpaca, vicuna and silk as well as inorganic fibers, such as asbestos, which is subject to much scrutiny because of toxicity.

The first man made fibers were regenerated cellulose (rayon), cellulose di and triacetate and regenerated protein (azlon). The first synthetic fibers were nylon, polyesters and PVC but these have been supplemented by acrylics, polyolefins, polyurethanes, and aromatic polyamide (aramids). Inorganic fibers, such as glass, metal fibers and ceramic fibers are also available [1].

Fiber Production

The fiber industry is a mature industry whose increase in production should parallel increases in population. However, as noted by Elias [2], the growth of the fiber industry, during the past forty years, has been 50 percent greater than the population growth [2].

The annual world wide production of natural fibers and that of manmade fibers is approximately 16 million tons. Rayon and acetate rayon account for 3 million tons. The annual consumption of synthetic fibers in millions of tons in the U.S., ECC, Japan and other countries is 3.2, 2.6, 1.4, and 6.3, respectively [3].

Polyester is the major synthetic fiber, but as shown in the following table, sizable quantities of other synthetic fibers are also consumed in the U.S., Western Europe, and Japan.

ANNUAL CONSUMPTION OF SYNTHETIC FIBERS (Million Tons)			
	U.S.	ECC	Japan[4]
Polyester	1.5	0.85	40.62
Polyamides (nylons)	1.1	0.64	0.26
Acrylics	0.29	0.8	0.4
Polypropylene etc.	0.8	-	-

American mills used 1.3 million tons of cotton in 1985 and this accounted for 30% of all fibers processed. The U.S. Government compensates cotton growers so that they are able to sell cotton competitively in world markets [5]. A new multifiber arrangement (MFA-4) effective until 1999 has been made with 54 countries which export cotton, abaca, coir, henequen, jute, ramie and sisal.

The rate of imports of synthetic fibers by U.S.A. has increased by 14 percent and the rate of exports has decreased by 13 percent [6].

Cellulosics

A process for the preparation of amorphous dietary cellulose from nonwoody plants has been developed by the U.S. Department of Agriculture [7]. 75 million tons of pulp and paper board were produced in the U.S. in 1986. This volume included 6 million tons of coated paper.

Because of the waste disposal problem, attempts have been made to replace or modify the kraft or sulfite pulping process. In one of these alternatives, developed accidentally by the son of the inventor, Dr. Young, ethyl acetate is added to a dilute solution of acetic acid and this "ester process" removes lignin from the wood and leaves a 60-70% yield of high quality pulp [8]. In another alternative process, oxygen or ozone is added to a caustic soda-anthraquinone pulping process at 160°C and the wood is defiberized in this pulping process in the presence of magnesium salts which inhibit the degradation of the cellulose.

There are more than 10 lignin degrading enzymes in the white rot fungus but these are not very active and not available in large quantities. The Institut National-de la Recherche Agronemique (INRA) uses an improved ligninase (Phanerochate chrysisperium) with hydrogen peroxide to obtain purified cellulose. The USDA has funded a program to investigate ligninase degradation of wood at its Forest Products Laboratories in Madison, WI and is seeking additional financial support from paper companies [9].

Vigo of USDA has produced a fiber which absorbs thermal energy when heated and releases this energy when cooled by crosslinking cellulose fibers with polyethylene glycol and urea resins in the presence of acids. This reversible thermal effect is based on a phase change in crystalline structure [10,11]. Over 60 thousand tons of soluble cellulosics are used annually by the U.S. pulp and paper industry [12].

Asbestos

Over 225 thousand tons of chrysotile asbestos was consumed in the U.S. in 1984 [13]. Because asbestos has been identified as a carcinogen, its use is restricted to applications where no economical substitute is available. Polytetrafluorethylene (PTFE) and flexible graphite have been used in place of asbestos in gaskets. The graphite is suitable for high temperature applications (2750°C) in the absence of oxygen but only at 425°C in the presence of oxygen. PTFE has poor resistance to creep but this deficiency can be overcome by the incorporation of fillers [14]. Aramids, ceramics and steel have also been considered as replacements for asbestos [15].

A sharp reduction in the toxicity of asbestos has been observed when it is heated at 120°C in the presence of phosphorus oxychloride vapors. No increase in cancer, tumors or lesions was observed in a $2.5 million study at Los Alamos National Laboratory when animals were exposed to extremely high concentrations of asbestos. According to Dr. M. F. Stark, the size and shape of the asbestos particles and not the asbestos are responsible for adverse health effects in asbestos environments.

Inorganic Fibers

Fiber glass, which is produced by the melt spinning of small glass beads is the only widely used inorganic fiber. Both blown glass fibers (staple) and continuous filament glass yarns, treated with coupling agents, are used as reinforcement for plastics. Glass yarns are also used as reinforcements in pneumatic tires. Fibers with high silica content are made by the acid leaching of glass fibers.

Inorganic fibers also being made by chemical vapor deposition (CVD) from boron, and silicon carbide. These fibers have been developed by AVCO and GTE. A matrix of silver nitride and silicon carbide whiskers is being injection molded for internation combustion engine parts. In some cases, the inorganic fibers are produced by the ignition and sintering of an extruded viscous-inorganic filament. Glass fibers are being rubber coated when used in alkaline environments [16].

Nylon-Aramids

Aliphatic-didactic nylons, which were the first synthetic fibers, monadic nylons and aromatic polyamides (aramids) are being used both as *textiles, reinforcements,* and molded plastics. Nylon-66 is made by the condensation of hexamethylenediamine and adipic acid and nylon-6 is produced by the self condensation of caprolactam.

The first aramid (Nomex) was polyphenyleneisophthalamide. Another aramid (Kevlar) which is poly-*p*-benzamide, is a nematic liquid crystal in sulfuric acid. Over 10,000 tons of Kevlar was produced in 1986.

The rate of spinning of nylon-66 has been increased by extrusion in an alkaline solution of cellulose. The strength and toughness of nylon-66 reinforced plastics has been improved by pultrusion of resin-impregnated continuous filaments [17]. Woven-aramid-epoxy resin laminates have less tendency to fail by delamination fracture [18].

The rapid growth of Nylon-66 fibers has been based on an increase in use of nylon carpets. Sales of intermediates for fiber production have remained unchanged in recent years [19]. A 750Km trip to the North Pole on May 1, 1986 by six member of the Steger International Polar Expedition would have been impossible without the use of nylon clothing.

Allied is promoting the use of nylon-66 (Stanyl) produced by DMS in the Netherlands. The aliphatic polyester fibers produced by Carothers in the early 1930's lacked thermal resistance and were replaced by melt-spinnable polyethylene terephthalate (PET). PET fibers are hydrophobic but blends with cotton and wool are hydrophilic and more "breathable" than cotton. Both nylon and polyester knitted or tufted fibers have