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Publisher: Taylor & Francis

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Polymer Reviews

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/lmsc20>

Recent Advances in Polymer Fibers

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Version of record first published: 02 May 2008.

To cite this article: Richard Kotek (2008): Recent Advances in Polymer Fibers, Polymer Reviews, 48:2, 221-229

To link to this article: <http://dx.doi.org/10.1080/15583720802020038>

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Perspective

Recent Advances in Polymer Fibers

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In recent years, there has been steady progress in developing new polymers and functional polymer fibers. The objectives of this issue are to provide readers an overview of significant advances for the production of high performance fibers such as Kevlar, PBO, Spectra and Dyneema fibers and to describe new super strong M5 fibers, highly elastic XLA™ fibers and self-crimping T-400, T-800, and other fibers. Additional goals are to present futuristic technologies such as shape memory fibers and compare them with innovative spandex fibers as well as to describe unique nanofibers from biopolymers by using novel electrospinning methods. Finally new, high performance poly(ethylene naphthalate) fibers will be reviewed.

Keywords polymer fibers, high performance fibers, shape memory fibers, eletrospinning, biopolymers, polyester fibers

1. Introduction

Fibers are elongated objects with a high axial ratio. This feature makes them very useful objects that can be utilized to make very functional materials for us human beings. As a result, billions of people use them every day. As a matter of fact, each of us would like to present ourselves in the best possible way by wearing the most appealing clothing that is made from the best possible fibers. Many of us need biodegradable sutures while undergoing surgery. All of us live in homes that require fibers for air and water filters. A handy fibrous wipe makes it easy to clean our kitchens. Indeed, the large variety of fibers creates an endless number of applications.

We use both natural and synthetic fibers. Natural fibers have been used since ancient times. Recently, new bamboo fibers¹ have been introduced to the market and are starting to be used widely. These fibers exhibit natural anti-microbial properties and can be used in many textile applications as well as in “green” composites. Cotton, silk, wool or flax (perhaps the oldest fiber in the world) have a strong presence in our daily life.

Interestingly, known fibers are polymers. Most of them are just linear macromolecules. We give credit to Dr. Staudinger, a Nobel prize winner, who was first to point out that

Received 23 February 2008; Accepted 25 February 2008.

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polymers are linear covalently bonded molecules and are not aggregates as previously believed. He laid the grounds for synthetic organic polymer and fiber chemistry. Soon after this discovery the pioneering work of Dr. Carothers at Du Pont and Dr. Schlack at BASF brought us nylon 6,6 and nylon 6 polymer fibers respectively. Later on, poly(ethylene terephthalate) (PET) manufacturing technology was developed by Whinfield and Dickson in 1946 and polyester staple fibers became available. Nylons and PET constitute major polymer fibers. Many other polymers were developed over the years and many new macromolecules are synthesized every day.

In the recent years there has been steady progress in developing new polymers and polymer fibers. Significant advances have been made in the production of high performance fibers, elastic fibers, and nanofibers from biopolymers by using a novel electrospinning method as well as high performance polyester fibers. As a result, the objective of this issue of *Polymer Reviews* is to bring the reader up to date and give an overview of these new developments.

2. High Performance Fibers

Recently, much effort has been concentrated on producing ultra-high modulus polymers.² The covalent bonds in these polymers are responsible for their high strength. However, manmade polymers generally do not exhibit the corresponding potential high modulus. High modulus and strength may result from structural perfection, such as straight, well aligned, stable, and densely-packed chains. A combination of extended chains and high crystal orientation is generally involved.

It is well known that the highest elastic moduli reported for linear polymers are generally much smaller than their theoretical values. Nakamae and coworkers³ measured "theoretical" elastic modulus, which was determined by observing strain-dependent X-ray diffraction in the polymer chain direction. This theoretical elastic modulus value was associated with the ultimate polymer modulus. Most polymers show tensile moduli far below those of their crystalline lattice in the chain direction. Only ultra drawn high molecular weight polyethylene (UHMW PE), isotactic polypropylene and Kevlar showed moduli close to the theoretical values. Polyamide fibers achieved maximum moduli only 1/20 of their theoretical value.

In the case of polymers with flexible backbone chains, strong and stiff polymer structure can be achieved by converting the flexible chains into highly oriented and extended chain conformations. The result is substantially increased tensile properties similar to ultra drawn high molecular weight polyethylene. The high modulus of polyethylene was obtained by solution spinning (gel spinning) with an ultra high draw ratio. Zachariades et al.⁴ successfully drew ultra high molecular weight polyethylene more than 200 times and obtained almost the theoretical modulus value at that draw ratio. Solution grown ultra high molecular weight polyethylene (UHMWPE) crystal morphologies were deformed to fibrillar structures at draw ratios more than 200. This high draw ratio is due to the smaller number of chain entanglements and inter- and intralamella tie molecules in their more regular, adjacent reentry chain-folded crystalline morphology.

High performance polyethylene fibers are currently commercially produced by gel spinning methods by DSM High Performance Fibers based in the Netherlands, by the Toyobo/DSM joint venture in Japan, and by Honeywell (formerly Allied Signal or Allied Fibers) in the USA. The strength of Spectra 1000 reaches a Young's modulus of 124 GPa and tenacity of 3.51 GPa. A lot of work has been done to improve thermal stability of these fibers as reported in this issue by Afshari and Lee.

Sikkema (the contributing author) and co-workers developed M5 polymer fibers. These are super strong fibers with a modulus of 330 GPa and tenacity of 5 GPa.

Du Pont de Nemours is currently developing commercial M5 fibers and yarns. A very interesting monomer namely, 2,5-dihydroxyterephthalic acid is used for making poly{2,6-diimidazo[4,5-b:4',5'-e]pyridinylene-1,4-(2,5-dihydroxy)phenylene} (PIPD). The unique feature of these polymers is that the two hydroxyl groups (on terephthalic acid) can form intermolecular hydrogen bonds and therefore fibrillation, which is often a problem for aramid fibers, is practically eliminated. As a result, M5 fibers have the highest compressive strength among synthetic fibers. Exploratory evaluation of the UV stability of M5 indicated excellent performance in that field. The mechanical properties of this new fiber make it competitive with carbon fiber in most applications—in light, slender, load-bearing, stiff, advanced-composite components and structures.

Tremendous work has been done to develop super strong Kevlar and, recently, PBO fibers. Recently DuPont de Nemours announced plans to expand production of Kevlar polymers at their Spruance plant by 25 percent by 2010 in order to meet growing demand.⁵ Due to its high tenacity, high energy dissipation, low density and weight reduction, and comfort, Kevlar is incorporated in bullet proof vests, helmets, property protection, panels, vehicle protection, and strategic equipment shielding to protect human life.

PBO fibers were commercialized by Toyobo Co. in 1998 under the trade name Zylon after about 20 years of research in the United States and Japan. PBO fiber has outstanding tensile modulus (352 GPa) and tensile strength (5.6 GPa) compared to other commercially available high performance fibers. Its specific strength and specific modulus are 9 and 9.4 times that of steel.^{6,7} Unfortunately for PBO, with great performance came great problems. The resistance of PBO to UV light and visible radiation is notoriously poor. PBO also lacks axial compressive strength. The tensile strength of PBO fiber also reduces in hot and humid environments. Considerable effort has been devoted to chemical modifications of PBO fiber to enhance axial compressive strength.

Both Kevlar and PBO fibers are reviewed by Afshari and coworkers in this issue. Other high performance products such Vectran or PVA fibers (Kurray) will not be covered. We hope to assemble another issue on specialty synthetic fibers in the near future.

3. Elastic Fibers

A review of elastic fibers in this issue is presented by Professor Hu and co-workers of the Hong Kong Polytechnic University.

A number of companies produce a variety of elastic fiber that exhibit elasticity and reversibility. They can be obtained by spinning polymers of specific molecular structure or modified polymers. On the elastic elongation basis, elastic fibers can be classified by high elastic fiber (elongation of 400–800%), medium elastic fiber (150–390%), low elastic fiber (20–150%), and micro-elastic fibers with elastic elongation below 20%.⁸

Traditional elastic fibers such as spandex or Lycra are well known segmented polyurethane fibers that are commercially produced via a dry spinning method. However, many new elastic products have been developed that include highly hydroscopic and moisture liberating spandex (by Asahi Kasei) or highly soft spandex to name a few.

Easy setting spandex is another very interesting product that can be heatset with PET fibers. The thermal stability of polyester spandex is poor, so it cannot be interwoven with polyester fiber. Asahi Kasei has developed a low temperature setting spandex named Roica BX, which not only has good setting but can interweave with polyester fiber and set under high temperature.

Another innovation is self-crimping elastic fibers. Du Pont de Nemours (Wilmington, DE) started to study the first self-crimp yarn (PP) in the early 1960s. Recently, the newly

commercialized self-crimp products of Du Pont, polyester T-400 and nylon T-800, have become very popular in the market. Unitica (Hyogo, Japan) also commercialized the self-crimp yarns, Z-10 and S-10. Furthermore, a nylon/polyurethane bicomponent filament, Sideria, developed by Kanebo (Japan), can adapt heat treatment to self-crimp itself to an appropriate degree.

XLATM is an olefin-based stretch fiber that is naturally resistant to harsh chemicals, high heat, and UV light and offers performance advantages compared to existing elastic fibers. This very new and exciting technology comes from Dow Chemical and is presented by Casey, our contributing author.

Incorporating XLA fiber into fabrics offers unmatched opportunities for developing easy-to-handle, durable garments with improved shape retention. In the USA we see Lastol fiber, the new generic name for this polyolefin based elastic fiber.^{10–13} The special microstructure of XLA combines long, flexible chains with crystallites and covalent bonds or cross-links, forming an intricate network. Using Dow proprietary electron beam cross-linking technology, the length of the chains and the number of crystallites are specifically controlled to give XLA fiber its unique elastic profile. High stretch is achieved with low levels of force, allowing garments to stretch and flex effortlessly and still return to their original shape.

Another futuristic technology is shape memory fibers. As Professor Hu points out: “The future aim is to investigate two-way multi-stimulus, multi-function bionic shape memory polymers, which can be activated by thermal, humidity, chemical, magnetism, and electricity or optical stimulus and has anti-bacterial, antistatic, anti-mildew, or ultra-violet resistant functions and to establish systematic, comprehensive and integrated theory of the shape memory polymer and apply the shape memory polymer in textiles”. It will not be long before these ideas become reality in our laboratories and commercial plants.

4. Electrospun Fibrous Materials

Conventional fiber spinning techniques such as wet spinning, dry spinning, melt spinning, and gel spinning can produce polymer fibers with diameters down to the micrometer range. If fiber diameter is reduced from micrometers to nanometers, very large surface area to volume ratios can be obtained. These unique qualities make polymer nanofibers an optimal candidate for many important applications.¹⁴ Polymer fibers can be generated from an electrostatically driven jet of polymer solution or polymer melt (Fig 1). This process, known as electrospinning, has received a great deal of attention in the last decade because of its ability to consistently generate polymer fibers that range from 50 to 500 nm in diameter.^{15–19} Because of the small pore size and high surface area inherent in electrospun textiles, these fabrics show promise for exploitation in soldier protective clothing (to help maximize the survivability, sustainability, and combat effectiveness of the individual soldier system against extreme weather conditions, ballistics, nuclear, biological and chemical warfare). Filtrations, membrane, reinforcing fibers in composite materials, optical and electronic applications (piezoelectric, optical sensor) are other fields where they could be of potential application. Drug delivery with polymer nanofibers is based on the principle that the dissolution rate of a particulate drug increases with increasing surface area of both the drug and the corresponding carrier. Many biomedical devices of practical use (cosmetics: skin healing and skin cleansing, wound dressing, drug delivery, and pharmaceuticals) can be fabricated with nanofibers. They could also be used as supports for enzymes or catalysts and scaffold for tissue engineering and templates for the formation of hollow fibers with inner diameters in the nanometer range.^{20–27}

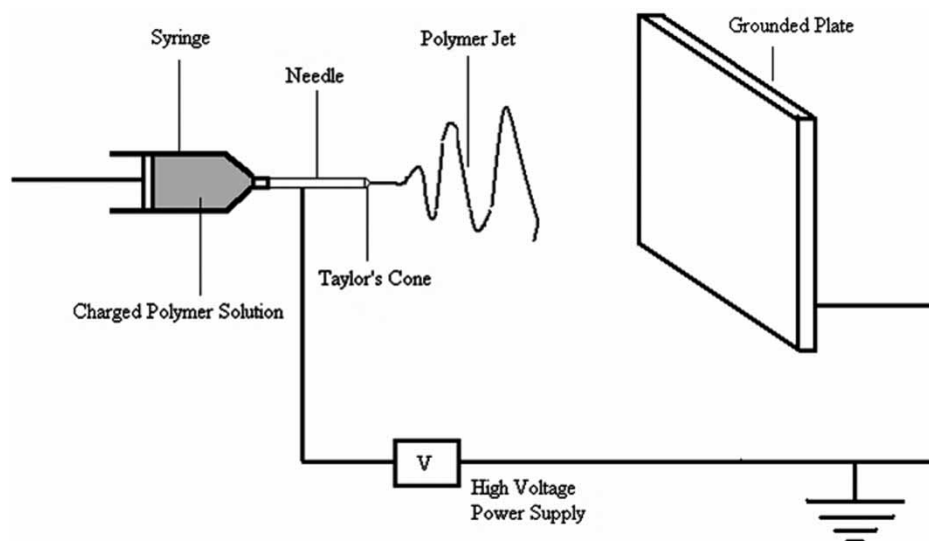


Figure 1. A schematic of electrospinning process.

In principle, electrospinning produces a mat of fibers. This is a very slow but steady process if only one jet is used. It resembles the work of the spider that can produce a very sophisticated web overnight. Commercial productions of electrospun web have existed since the 1980s but little information was shared with the public.

Several years ago a group of scientists from the Technical University of Liberec (TUL) demonstrated NanospiderTM technology, a variation on the electrospinning process, that uses a cylinder instead of nozzles (Fig. 2).²⁸ The machine can produce nanofibers weighing between 0.1 and 10 g/m² in diameters ranging from 200 to 500 nm. Nanospider technology can produce 0.1 to 1 g of material in less than one minute. By comparison, it would take as much as one hour to achieve the same result using traditional electrospinning techniques.²⁹ Elmarco (Liberec, Czech Republic) currently offers commercial high capacity and laboratory machines for the production of nanowebs.

Schiffman and Schauer describe biopolymers as renewable resources³⁰ which also intrinsically exhibit antibacterial activity, biodegradability, and biocompatibility.³¹ These factors made us review biopolymers as materials for electrospinning. Professor Schauer's review is very detailed with over 280 references and therefore will be an excellent resource for a scientist working in the field. An interesting aspect in her work is the focus on the numerous applications of nanowebs in medicine and many other fields. Biopolymers will include polysaccharides (cellulose, chitin, chitosan, dextrose), proteins (collagen, gelatin, silk, etc), DNA, as well as some biopolymer derivatives and composites.

Due to very high surface area to volume ratios and high porosity with an interconnected pore network, polymeric nanofibrous structures are being explored with interest in a broad range of applications^{32,33} from scaffolds for tissue engineering to products for filtration and protective clothing and many other applications.

In this issue, Moghe and Professor Gupta provide a very innovative approach for making nanofibers in a core-sheath configuration, by using two dissimilar materials, via the novel technique of co-axial electrospinning. This approach offers unusual potential for use in many novel applications. The studies have addressed issues related to the



Figure 2. Rotating electrode²⁹ the essence of Nanospider TechnologyTM for making commercial nanofibers (reprinted with permission from Nanopeutics).

technology involved and examined the suitability of the technique for producing unique nanoscale morphologies involving a variety of materials. In this first major review of co-axial electrospinning, they provide details of the manufacturing and material factors affecting the process, the conditions needed for preparing desired uniform morphologies, and the different types of structures that have been successfully produced.

Cellulose, which is found in plant walls, is the most abundant raw material on earth. Millions of pounds of this biorenewable polymer are produced every year. The total consumption world wide of cellulosic fibers in 1998 was 4,817 million pounds. It is plentiful, inexpensive, and biodegradable. It is capable of producing a number of fibrous products with excellent properties whose utility extends into numerous end uses and industries. Cellulose is an excellent source of textile fibers, for both the commodity and high-end, fashion-oriented markets. A common example is rayon. In addition, cellulose provides fibers for industrial end uses requiring strong, tough fibers. A common example is fibers used in tire cord.

In recent years cellulose dissolution has been researched quite extensively and new solvents have been discovered that are more environmentally friendly. Several new processes that rely on these solvents have been developed for manufacturing fibers.^{34,35} Furthermore, research has also been focused on cellulose derivatization processes that pollute less and are more economical. Professor Frey brings her expertise in cellulose and reviewed cellulose electrospun fibers to these topics.

5. New Polyester Fibers

Polyesters, especially poly(ethylene terephthalate) (PET) were manufactured as industrial products by ICI (UK, 1949) and Du Pont (USA, 1953) soon after the technology of manufacturing was developed by Whinfield and Dickson in 1946. PET or commonly named polyester fibers are extremely functional materials particularly for blends with cotton fibers. In the past decade some other improved and useful polyesters have also been developed.

An interest in biodegradable polymers and progress in biotechnology have led to the commercial production of lactic acid and poly(lactic acid) (PLA) by Cargill Dow LLC in 2002.³⁶ PLA is a low melting, thermoplastic polymer and can be easily processed by melt spinning. Therefore it sparked a lot of interest in making non-woven biodegradable materials and textile fibers. The polymer also dissolves in some organic solvents so other spinning techniques such as wet spinning, dry spinning, or electrospinning could be employed. PLA fiber is useful for making biodegradable textiles. Numerous medical applications such as sutures, pharmaceuticals, and tissue engineering have been explored.

Poly(trimethylene terephthalate) (PTT) is a polyester made by the polycondensation of 1,3-propanediol (PDO) with terephthalic acid. Although, first synthesized by Whinfield and Dickson in 1941, PTT remained an obscure polymer because one of its raw materials, 1,3-propanediol, was not readily available at the time. Recently, Shell Chemical has commercialized a route based on ethylene oxide and carbon monoxide. 1,3-propanediol is also being produced by DuPont Tate and Lyle Bioproducts, a joint venture between DuPont and Tate and Lyle, in Loudon, Tennessee. Dubbed Bio-PDO, it is being produced with a genetically modified strain of *E. coli* that's fed a refined corn syrup. The bacteria, after a fermentation process, then produces Bio-PDO. Most in the industry refer to Bio-PDO as "Liquid Diamonds", due to the crystal clear liquid that is the final product.³⁷ Both companies Shell and Du Pont produce PTT. Du Pont de Nemours has a continuous production of PTT in Kinston, NC. PTT is more expensive than PET, but becomes a very useful textile fiber. Because of its low melting point of 223°C, the polymer can be melt spun at lower temperatures than PET. Furthermore PTT fibers can be dyed with dispersed dyes at atmospheric pressure with excellent dye exhaustion and color fastness.³⁸

Recently, with the availability of monomers for poly(ethylene naphthalate) (PEN) at large scale and low cost, much attention has been paid to develop PEN fibers because its superior mechanical and thermal properties can compete with the most commercially important poly(ethylene terephthalate) fibers, especially in some performance-driven markets. Chen and Professor Kotek review technical papers, product reports, and patents for the production of PEN fibers via melt-spinning. The mechanical properties of PEN fibers will also be presented as a function of spinning and drawing parameters as well as the morphology of PEN polymer fibers. Up to now, PEN fibers have been commercially available from several producers including Teijin in Japan, Performance Fibers in the USA and Europe, Kosa in Europe, and Hyosung in Korea.

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