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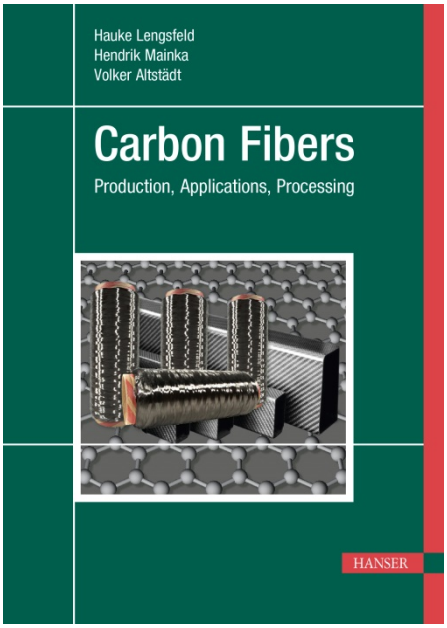

Sample Pages

Carbon Fibers

Hauke Lengsfeld et al.

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Volker Altstädt undertook university studies in physics and received a Dr.-Ing. in 1987 with Prof. Dr.-Ing. Ehrenstein at the Department of Mechanical Engineering at the University of Kassel (Germany). For eight years he was working at the Department for Polymer Composites and later for Polymer Physics at BASF AG in Ludwigshafen. Since 1995 he has been professor for Plastics in Mechanical Engineering and Head of the Department of Polymers and Plastics Composites at the Technical University of Hamburg-Harburg. Since October 2000 he has been full professor and Director of the Department of Polymer Engineering at the University of Bayreuth. In summer 2009 he became in addition the CEO of the Bavarian Research Company NMB GmbH in Bayreuth.

Preface

According to the composite market report of CCeV and AVK, a global carbon fiber market of about 70.5 thousand tons was identified for 2017 [1]. Mordor Intelligence expects an annual growth rate of 13.45% for carbon fibers for the period 2018 to 2023. Compared to other fibers such as glass fibers with several million production tons/year at a growth rate of approx. 7.4% [3], the consumption of carbon fibers worldwide is very low but the volume growth is very high. On the other hand, the applications are highly innovative and mostly driven by the goal of weight reduction. Demand is highest in the aerospace industry (36%) and the automotive industry (24%). But also the wind energy and sports industries with 13% each and the construction industry (5%) are important sales markets [2].

Against this background, the aim of this technical book is to present for the first time a book on fiber composite technology with a focus on modern carbon fibers. The focus is on the individual characteristics of carbon fibers, from the precursor for the fiber, the stabilization and carbonization process, to the resulting atomic structure and the properties. The various paths from the fiber to the roving to the composite material and its properties are discussed using many current examples from various branches of industry.

After a brief introduction to the historical development of carbon fibers, the *second chapter* of the book first explains the atomic model for carbon compounds in order to make the various carbon modifications (diamond, C-nanofiber, fullerenes …) comprehensible to the reader.

The *third chapter* describes in great detail the process steps of precursor production, oxidative stabilization, and carbonization/graphitization. The discussion of surface treatment and the correct sizing of the fibers is also important for the reader. Very instructive are the many graphic representations, e. g. of C-fibers with and without sizing, as well as the ideas about the interphase.

This chapter is logically followed in *Chapter 4* with the analysis of carbon fibers and rovings made from them. In addition to a presentation of the relevant standards, current test procedures for characterization and classification of the processing behavior of carbon fiber yarns and filaments are addressed. Test apparatuses are

clearly illustrated in pictures and, for example, drapery faults caused by forming are clearly explained by means of photos.

A particular strength of the book is the presentation of the applications of carbon fibers in *Chapter 5*. Due to the excellent and diverse properties of PAN-based fibers, they are used in a wide variety of applications. The book gives application examples of the different fiber types (PREOX and carbon fibers) and explains with examples the different presentations (continuous, cut, ground) from a practical perspective and with rich illustration, to a degree that can hardly be found in other sources. The presentation is subdivided according to the different fiber types and covers the broad spectrum of production methods for applications in the aerospace industry, from automotive structures made of CFRP based on multiaxial fabrics to PrimeTex fabrics for winter sports skis, carbon short fibers for screed concrete or in a 3D-printed fan housing, to name just a few examples.

Since C-fiber production is very energy-intensive, it is no longer possible to talk about materials today without addressing their recycling and carbon footprint in the lifecycle analysis. *Chapter 6* is therefore devoted to a current review of C-fiber recycling and sustainability. Here, too, a distinction is made between the different presentations (continuous, cut, ground) in the description of the various recycling paths. In addition to the actual recycling cycle for C-fibers, the challenges of waste treatment are also addressed and ways are shown of moving from a material mix of different residual materials as an input variable to an energetic or better recyclate semi-finished product (flour, balls, or granulate) by thermal, chemical, or mechanical processes, which lead to sustainable secondary use in a variety of applications shown in the book. The authors come to the conclusion that the recycling of all types of carbon-containing waste and the associated recovery of high-quality carbon fibers (rCF) has been solved on an industrial scale and is increasingly applied.

Nevertheless, there are still challenges to be met in order to be able to expand the use of carbon fibers. These are mainly driven by the high price of the fiber. In *Chapter 7,* precursor costs, energy costs, and investment costs for plants are subdivided into future improvement potentials.

In summary, this technical book can be used both as a manual and as a textbook or reference book for practitioners. The physical basics of carbon fibers, the manufacturing processes, their material, and process engineering basics are taught with a strong practical orientation. The book also aims to make a contribution at the interfaces between carbon fiber technology and semi-finished products and components made from them, and to build the necessary bridges between structure/production and properties. Looking to the future, the book presents important developments in the field of recycling and energy efficiency of modern carbon fiber composite technology.

Bayreuth, February 2019 *Volker Altstädt*

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[2] Study of the Carbon Global Fiber Market – *https://www.mordorintelligence.com/industry-reports/global-carbon-fiber-market-industry*

[3] *https://www.reportlinker.com/p05176097/Global-Glass-Fiber-Market.html*

Contents

1 Introduction

Uniqueness

The uniqueness of carbon fibers is defined by their excellent properties. Their strength is comparable to that of high-performance steel and their stiffness is higher than that of all known metals, ceramics, or polymers. Even their thermal and electrical conductivity exceeds that of comparable materials (see Chapter 3). The uniqueness of the properties of carbon fibers becomes apparent when the strength or stiffness is divided by the material density to obtain the specific properties. No other known material can currently compete with this result [1–1].

History

Carbon fibers are a synthetic product. The history of carbon fiber began in 1879 with the invention of the first filament lamp by *Thomas Edison*. Edison experimented with fine cotton and bamboo fibers in order to improve the already known arc lamp. He pyrolyzed (carbonized) the fibers at high temperatures. The resulting carbon filaments showed good electrical conductivity and stability for use in incandescent lamps. Unfortunately this application for carbon fiber was soon replaced by tungsten filaments for this application due to their easier and more cost effective manufacturing process. As the result of this development, the initially interest in carbon fiber slowed down again [1–2].

The modern era of carbon fibers did not begin until the 1950s and 1960s. Fundamental development work was done on characterizing the first carbon fibers based on polyacrylonitrile (PAN), pitch, viscose, and rayon in the development laboratories of *Union Carbide* and *DuPont*. It was discovered that pyrolysis of carbon-rich materials resulted in an oriented graphite structure, with a very high modulus of elasticity and strength values. This discovery could be demonstrated in practical tests on so-called graphite whiskers. Whiskers are thread-shaped single crystals with an anisotropic lattice structure [1–3]. Soon several researchers independently recognized the necessity of stretching the fiber at high temperatures (> 2800 °C) in order to further improve the properties, especially the modulus of elasticity. This allowed up to 10 times higher stiffnesses to be achieved than without the stretch-

ing process. One of the first commercial fibers from Union Carbide was the "Thornel-25". This trade name derives from the Nordic deity *Thor* ("strength").

At the beginning of the 1960s, Japanese scientists were able to successfully demonstrate in a study the production of high modulus or high stiffness fibers based on polyacrylonitrile (PAN) as a precursor. Earlier attempts of US-American scientists had failed here. The secret of this development was improved PAN precursors. The chemical structure (the so-called backbone) after carbonization was the reason for their excellent properties. At the same time, British scientists also succeeded in developing high-modulus PAN-based fibers, which were soon commercialized. All these developments created the basis for the carbon fibers used today. Through increasing application and further development, production costs have been drastically reduced in the past 35 years through new processes and new raw materials [1–4].

Since around 2010, research and subsequent commercialization have focused on new precursors such as lignin and textile PAN on the one hand, as well as measures for energy efficiency on the other (plasma oxidation, microwave oven) [1–5]. The commercialization of these new technologies is driven by innovative start-ups such as *LeMond Composites, RMX,* and *4M* [1–6] [1–7] [1–8].

Current Applications

Due to their outstanding properties, carbon fibers are used in a wide variety of industrial sectors. Examples range from silicon carbide brake disks for aircraft and sports cars, tanks for solid-fuel rockets for space travel, hydrogen tanks and structural components for automobiles, to applications in the construction industry and sporting goods. These and other examples are discussed in detail in Chapter 5.

Recycling

Waste containing carbon fibers occurs in various stages during the production, processing, and use of carbon fibers and carbon fiber reinforced polymer (CFRP) components made from them. Recycling processes include pyrolysis, fluidized bed, solvolysis, and direct chemical use. The challenges that arise and the benefits that can be achieved are described in detail in Chapter 6.

Future Trends

Future applications of carbon fibers will be in the area of additive manufacturing (e. g., Big Area Additive Manufacturing), but also widespread applications in automotive and consumer-oriented areas seem possible if the prices for carbon fibers continue to fall as predicted. The most important requirement for this will be the successful commercialization of new precursors and conversion technologies for the production of carbon fibers.

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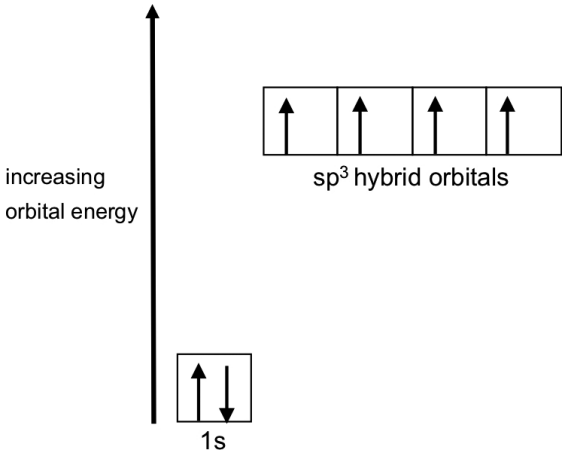

Figure 2.5 sp^3-hybrid orbitals of the carbon atom [2–2]

These orbitals are called hybrid orbitals and are responsible for the different modifications and structural forms of carbon, which are explained below.

■ 2.3 Structural Forms of Carbon

Diamond and graphite are the two main elemental structural forms (allotropes) of carbon as infinite solid carbon molecules.

In diamond, each carbon atom is sp^3-hybridized and surrounded by four more carbon atoms. All bonds in the diamond are covalent bonds and have a bond length of 0.154 nm. Due to the sp^3-hybridization, the basic shape of the diamond structure is a tetrahedron in which the carbon atoms are arranged.

In graphite, the carbon is sp^2-hybridized, resulting in a planar structure in which each carbon atom is connected to three other carbon atoms. The three covalent bonds have a bond length of 0.1415 nm. The individual layers of graphite are relatively weakly bound to each other. This attraction is called *van der Waals* forces.

In 1985 a new form of carbon was discovered with the fullerenes, which contain just sp^2-hybridized carbon. The relationship between the individual structural forms of carbon is summarized in Figure 2.6 [2–14].

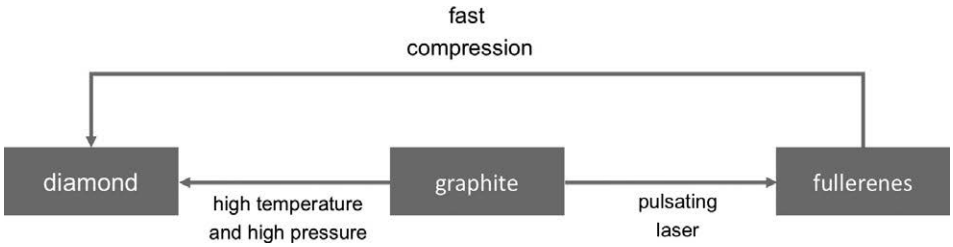

Figure 2.6 Allotropes of carbon and their interconversion

2.3.1 Diamond – The Diamond Structure (sp^3)

In diamonds, the carbon atoms are linked three-dimensionally by covalent bonds. Each carbon atom is tetrahedrally surrounded by four neighboring carbon atoms. It is bound to its neighbors by four σ bonds. The C–C single bonds (length: 1.5445 Å) are formed by overlapping tetrahedrally aligned sp^3-hybrid orbitals; see Figure 2.7.

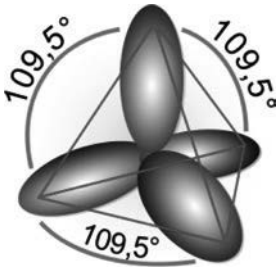

Figure 2.7
Four sp^3-orbitals – tetrahedral at equal angles [2–12]

2.3.2 Graphite – The Graphite Structure (sp^2)

Graphite is composed of flat carbon layers stacked on top of each other.

Within the layers, each carbon atom is surrounded by three carbon atoms in the form of an equilateral triangle. Each carbon atom is sp^2-hybridized and with three of its four outer electrons forms three localized σ bonds to its three atomic neighbors. The fourth valence electrons of the carbon atoms are located in delocalized π molecular orbitals, which result from a combination of the p atomic orbitals of the carbon atoms, which are not involved in hybridization and are perpendicular to the sp^2-hybrid orbitals. Thus, the carbon atoms of graphite are linked by σ bonds as well as by π bonds. In graphite, all C–C bonds have the same length.

3 Carbon Fibers

A carbon fiber is an endlessly long, very thin material strand with a diameter of approximately 5 to 10 μm, essentially consisting of pure carbon. The carbon atoms are arranged in a graphite structure largely parallel to the fiber axis, which makes for the high stability of the fiber [3–1]. Taken together, many thousands of these carbon filaments form a fiber bundle, also known as roving or tow.

The base and thus the starting material of each carbon fiber is its precursor. Since the early work of *Thomas Edison* and the use of cellulose-based carbon fibers in light bulbs, a variety of precursors for the production of carbon fibers have been tested [3–2]. Today there are two commercially available precursors: *polyacrylonitrile (PAN)* and *pitch*.

- PAN-based carbon fibers dominate the market and represent more than 90% of the world market. This is mainly due to the good availability of the precursor PAN and its constant quality.
- The remaining approximately 10% of the world market is occupied by pitch-based carbon fibers. In addition to the classical PAN- and pitch-based precursors, there are some promising new approaches to obtain suitable starting compounds on a different chemical basis (see Chapter 7).

Heat treatment is the conventional process by which carbon fibers are produced today. Figure 3.1 schematically shows the multi-stage production process for carbon fibers based on polyacrylonitrile – a process that contains both chemical and mechanical (stretching of the fibers) components. Three characteristic process steps are performed:

1. Precursor manufacturing
2. Oxidative stabilization
3. Carbonization/graphitization

The scheme gives an idea of how complex and cost-intensive the production of carbon fibers is, especially through energy-intensive heat treatment. This is a quasi-continuous process with several weeks of production time for one production lot.

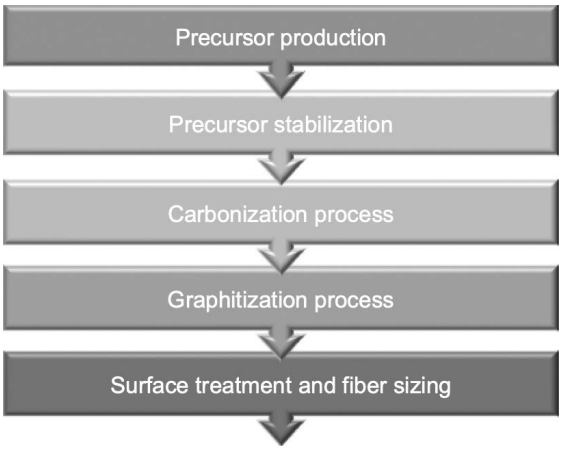

Figure 3.1
Schematic diagram of the manufacturing process for carbon fibers

After the first carbonization, so-called standard fibers or high-strength fibers (HS and HT types) are obtained. For high modulus fibers (IM, HM, UHM types) another step follows, graphitization. At the end of the production process, the carbon fiber obtained consists of > 95% pure carbon.

■ 3.1 Precursor Manufacturing

Suitable precursors are a fundamental prerequisite for the production of carbon fibers. These are long-chain, organic polymers with characteristic properties that significantly determine the later properties of the carbon fiber obtained from them. The exact chemical composition of precursors varies from manufacturer to manufacturer and is usually a strictly guarded secret, as the development of a new fiber type can take 10 years and more to develop. Most carbon fiber manufacturers also produce their own precursors or several different ones, depending on the product range. Typical variable influencing parameters in precursor manufacture are the solvents used, types and quantities of co-monomers used, degree of precursor yarn shrinkage, and yarn stretching in the process.

A precursor should be as economic and ecologically friendly as possible, since on the way to carbon fiber about 50% of the precursor's initial mass is lost as gaseous by-products as a result of the chemical conversion processes. An ideal precursor for carbon fibers already has a high carbon content and can be spun into a thin precursor yarn, mechanically stretched, and then converted into a carbon fiber. The fineness of the yarn is further increased by the mechanical stretching, i. e. the fiber diameter is reduced. The stretching also ensures an advantageous alignment

of the molecular chains along the fiber axis, which is important for the mechanical properties of the later carbon fiber.

There are two main processes for the production of a precursor filament: *wet spinning* and *melt spinning*. Wet spinning is the most commonly used commercial process for the production of PAN precursors [3–3][3–26]. This is due to the fact that the decomposition temperature of PAN is below its melting temperature. Although the melt spinning process was also developed for PAN, it has not yet been implemented on an industrial scale [3–4].

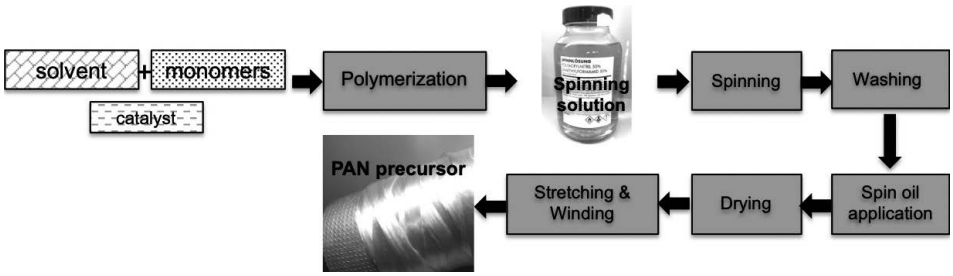

Figure 3.2 Process for precursor production

3.1.1 Wet Spinning

Figure 3.3 shows the typical process flow of a continuous solvent polymerization with subsequent wet spinning. This comprises the two essential steps of the so-called dope preparation (polymerization to polyacrylonitrile) and coagulation (spinning of the PAN). Dope is a general description for a polymer solution is an organic solvent ready for the spinning process (coagulation).

Dope Preparation

To produce dope, powdered acrylonitrile is reacted with other co-monomers, such as methyl acrylate and methyl methacrylate, using a catalyst dissolved in solvents such as dimethyl formamide (DMF) or dimethyl sulfoxide (DMSO) to form polyacrylonitrile. Instead of a solvent-supported reaction, it is also possible to convert the starting materials into a suspension. In order to prevent solidification of the mixture as a result of the polymerization reaction, the temperature of the polymerization reaction must be between 25 and 120 °C. The dope concentration is about 15 to 20% and is pumped directly into the spinning bath (coagulation) [3–5].

3.5.3 Manufacturing Capacities

Figure 3.34 provides an overview of the theoretical global production capacities for carbon fibers. These add up to around 151,000 tons, whereby the figures may vary depending on the source. Compared to the theoretical quantities of approximately 14,000 t [3–19] available in the year 2000, this means an over tenfold increase in capacities [3–20] [3–24].

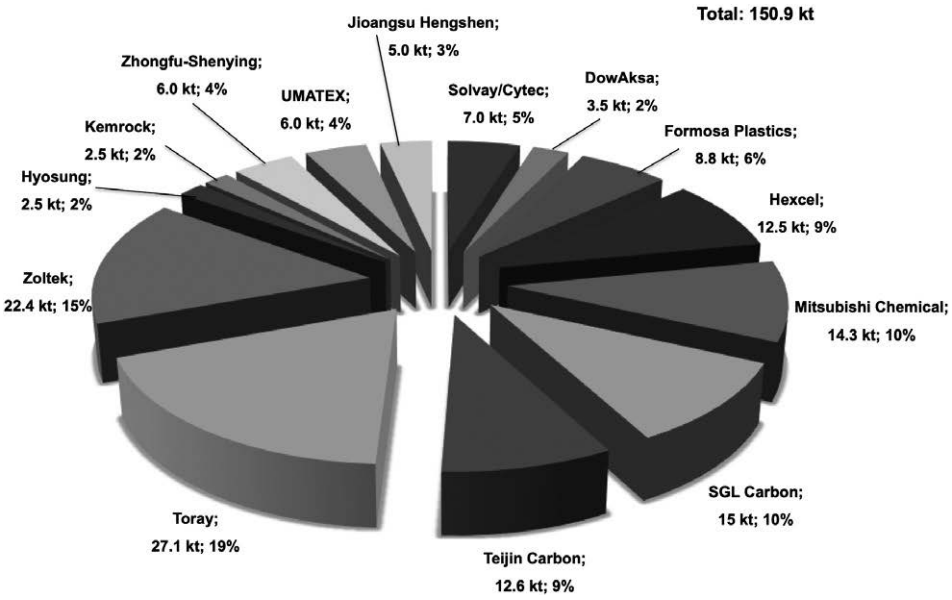

Figure 3.34 Annual theoretical production capacity for carbon fiber, as of 09/2019 (according to [3–20] [3–24])

These figures, based on manufacturer data, express only the theoretically producible quantity, not the actual world market demand for carbon fibers. No distinction is made between different fiber types, K number, or application areas (aerospace or industrial fibers). In Table 3.11 the absolute and percentage capacities per manufacturer are listed. Figure 3.35 illustrates the global distribution of capacity in a country comparison.

The chart in Figure 3.35 shows the theoretical production capacity broken down by country of manufacture. Three major regions account for more than 60% of capacity: North and Central America (38%), Japan (18%), and China (11%).

Table 3.11 Manufacturer, Brand Name, and Theoretical Manufacturing Capacities, as at 09/2019

Manufacturer	Type Designation	Capacity in kt	Capacity in %
Solvay (Cytec)	Thornel®	7.0	4.6
DowAksa	not specified	3.5	2.3
Formosa Plastics	Tairyfil	8.8	5.8
Hexcel	Magnamite, HexTow®	12.5	8.3
Mitsubishi Chemical	DIALED®, Pyrofil, Grafil	14.3	9.5
SGL Carbon	SIGRAFIL®	15.0	9.9
Tejin Carbon	Tenax®-E, Tenax®-J	12.6	8.3
TORAY	TORAYCA®	27.1	18.0
Zoltek	PANEX®	22.4	14.8
Hyosung	TANSOME®	2.5	1.7
Kemrock	JAITEC®	2.5	1.7
Zhongfu Shenying	SYT45	6.0	4.0
UMATEX	UMT®	6.0	4.0
Jiangsu Hengshen	not specified	5.0	3.3
LeMond Composites	GRAIL™	–	0.0
Other	not specified	5.7	3.8
	Total	**150.9**	**100**

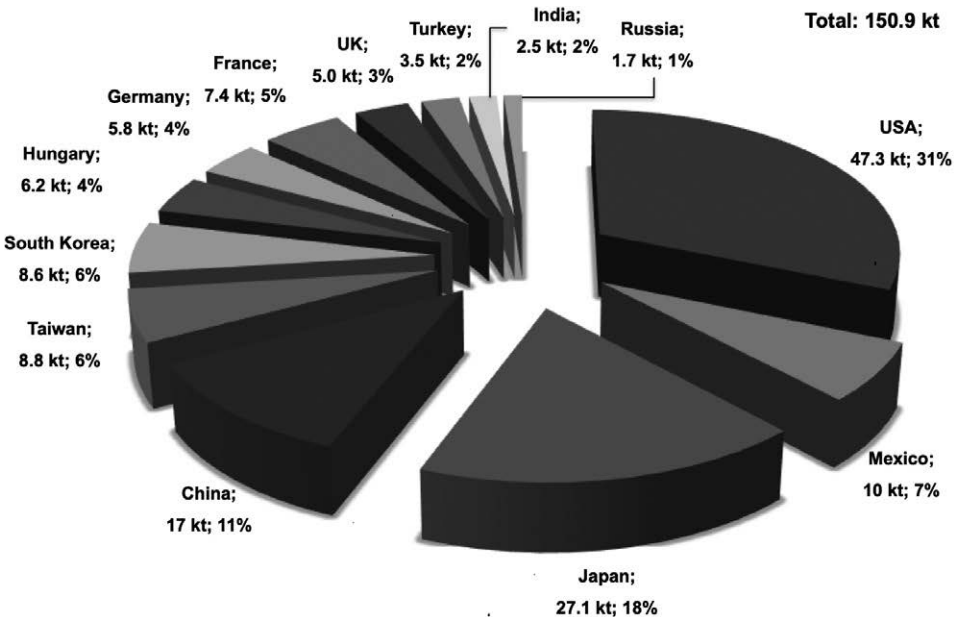

Figure 3.35 Theoretical production capacity by country of manufacture, as at 09/2019 (according to [3–20])

Method C (Density Gradient Column)

This method is based on the principle that a sample is immersed in a liquid column with a linear density gradient and does not sink or rise further within the liquid column according to the sample density. *Density gradient columns* are columns whose density increases uniformly from top to bottom. Thus, method C is basically based on the same principle as method B. First, a gradient column (height 1 m, ⌀ 40 to 50 mm) is produced from two liquids of different density. The density distribution in the column is determined using several calibrated reference bodies in the range of the expected carbon fiber sample density and plotted as a calibration curve. A quantity of 1 to 10 mg of the carbon fiber is dipped into the mixture from above in the form of a knot or bundle. The sample slowly sinks down in the column until an equilibrium is reached at the same density of fiber sample and liquid mixture and the fiber sample floats in the liquid. The density ρ of the sample in g/cm^3 results from the previously determined calibration curve of the gradient column.

Normative References:

- ISO 10119:2002: Carbon fibre – Determination of density
- ISO 10548:2002: Carbon fibre – Determination of size content
- ISO 291: Plastics – Standard atmospheres for conditioning and testing

4.1.2 Determination of Linear Density

Gravimetric Determination

The principle for determining the linear density is based on the gravimetric determination of a fiber bundle sample of defined and known length. The measurement can be carried out on carbon fiber yarn with or without preparation application (sizing). The sample is conditioned or dried under a standard climate before measurement. The sizing can be removed by solvent extraction, pyrolysis or chemical digestion. In this case, drying of the sample is not required.

At first the empty weight of a suitable bobbin is measured. Then it is wounded with a damage-free fiber sample of defined length. According to Table 4.3 sample length depends on the nominal linear density of the fiber.

Table 4.3 Overview Sample Lengths vs. Nominal Linear Density for Carbon Fibers

Nominal Linear Density T_f in tex	Sample Length in m
$T_f < 50$	Select length so that mass ≥ 0.25 g
$50 < T_f < 125$	5
$125 < T_f < 250$	2
$250 < T_f$	1

The mass m of the fiber sample is calculated from the difference between the two weighs (weight of empty spool or wound spool).

The linear density T_f in g/km of the fiber is obtained from the following equation and is expressed in tex:

$$T_f = \frac{1000 * m}{L}$$

m = mass of the sample in g
L = sample length in m

Vibration Method

Another method for determining linear density is based on the principle of vibroscopic measurement (ISO 1973). Figure 4.1 shows a machine developed by *Textechno GmbH* which largely automates the measurement [4–3]. For this purpose, a filament from a bundle of yarns is inserted into the measuring apparatus and vibrated by a sinusoidal frequency sweep via a loudspeaker. The linear density can be calculated from the resonant frequency f of the filament measured at a constant measuring length L and a known preload F_V according to Equation 4.2:

$$T_f = \frac{F_V}{4 * f^2 * L^2} \tag{4.2}$$

T_f = linear density
F_V = preload force
f = resonant frequency
L = free sample length

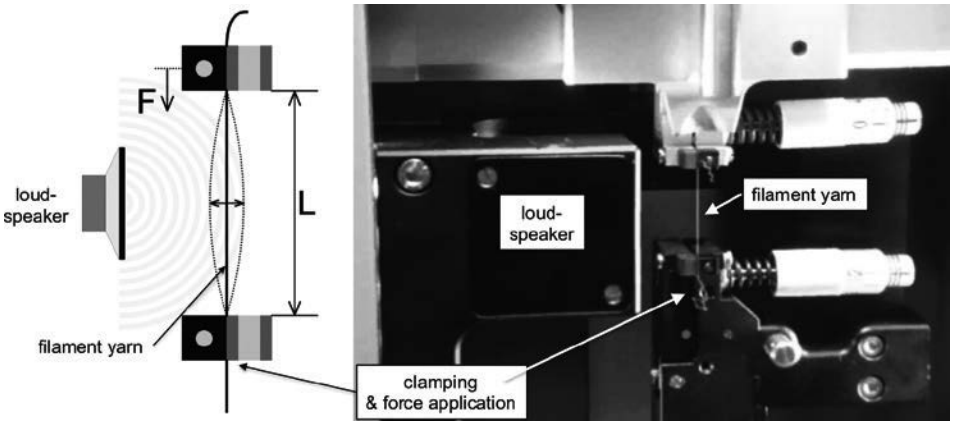

Figure 4.1 Measuring arrangement for vibroscopic determination of linear density [*Textechno GmbH*]

5 Applications of Carbon Fibers

5.1 Introductory Remarks

Due to their excellent and versatile properties, PAN-based fibers are used in a wide variety of applications. Application examples of different fiber types (PREOX and carbon fibers) and different fiber format (continuous, cut, and ground) will be explained in this chapter.

The diagram in Figure 5.1 shows a variety of dry and pre-impregnated fiber semi-finished products, starting with PAN fiber as precursor followed by PREOX fiber and carbon fiber. Furthermore, different possible processing technologies producing different end-products are presented.

Starting from a continuous PAN precursor fiber whose *K number* can be varied depending on the end application (see also Chapter 3), a so-called PREOX fiber (oxidatively stabilized PAN fiber) is produced as the first step in the carbon fiber production process. This PREOX fiber can either be directly converted into carbon fiber, or can be used in textile processing steps in the form of continuous or short cut fiber or fiber flour. The respective K number of the precursor depends mainly on the application of the fiber. It influences the property level and thus also the fiber price considerably. For example, while only low K numbers of 3 to 24 K are used for aerospace applications, yarn counts of 50 to 400 K are common for industrial and automotive applications.

Depending on the application, the fibers are further processed either as dry textiles (with subsequent matrix addition) or as semi-finished products pre-impregnated with matrix (e. g. thermoplastics). The diagram in Figure 5.1 shows how the wide range of semi-finished products can be processed into a finished component in a wide variety of ways.

The following examples show applications of carbon fibers and their processing into components and end-products.

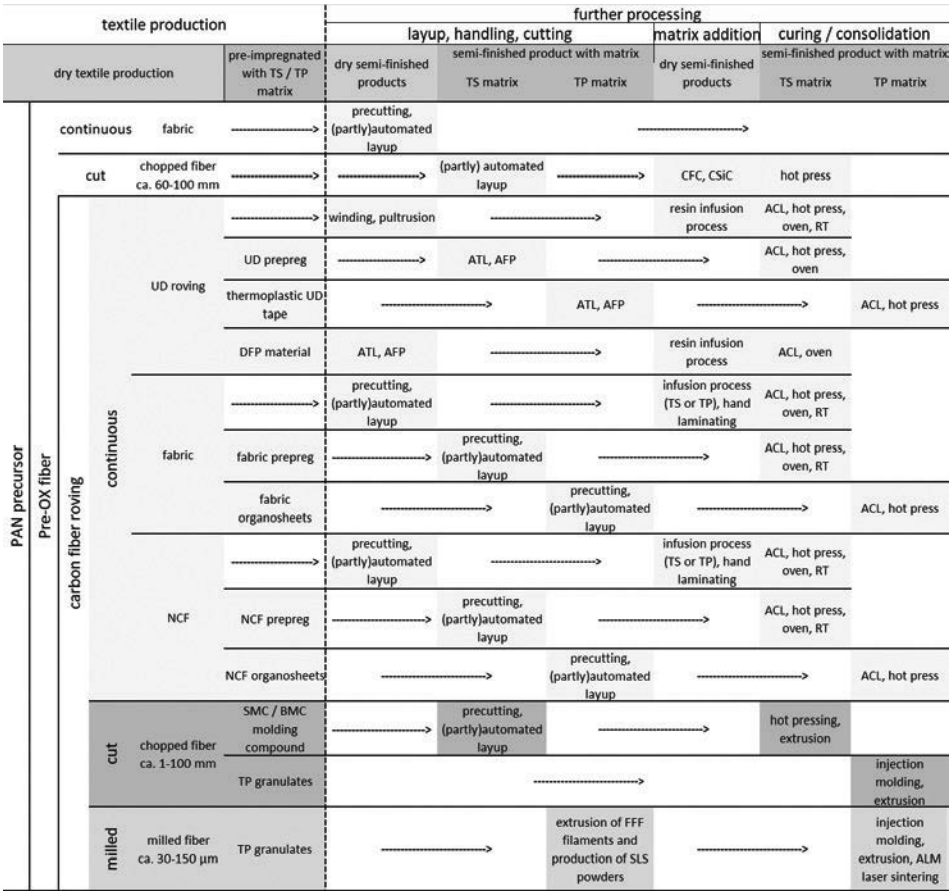

Figure 5.1 From precursor to application

■ 5.2 Dry Semi-Finished Fiber Products

5.2.1 PREOX Fibers

As described in Chapter 3, PREOX fibers or yarns are a thermally stabilized intermediate product in the carbon fiber production process. These can either be further processed into carbon fibers or used directly as continuous fibers or as crimped staple fibers. PREOX fiber types commonly offered on the market are summarized in Table 5.1.

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