Ceramic Matrix Composites

Fiber Reinforced Ceramics and their Applications

Edited by
Walter Krenkel

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Foreword

Ceramic Matrix Composites (CMCs) are *non-brittle* refractory materials designed for applications in severe environments (often combining high temperatures, high stress levels and corrosive atmospheres). Compared to other structural materials (such as steels, aluminium or titanium alloys, as well as nickel-based superalloys or monolithic ceramics), they are relatively new, still mostly at the development stage but with a few well-established and promising applications in different high technology domains. We will first summarize here the main features of this new class of materials, then show what impact they have (or could have) on the development of different high tech fields and mention some important historical milestones.

The high strength potential of CMCs is directly related to the use of high strength, high modulus ceramic *fiber reinforcements* of small diameter (typically of the order of 10 µm). Covalent non-oxide fibers, such as carbon or silicon carbide based fibers, are those which presently display the best mechanical properties at high temperature (particularly in terms of creep resistance), but they are oxidation-prone. In this field, the development in Japan of the *SiC-based fiber* family, from the pioneering work of S. Yajima in the mid-1970s, which exhibits better oxidation resistance than carbon fibers, has been an important milestone. Comparatively, refractory *oxide fibers* (such as alumina and alumina-based fibers) display, by their chemical nature, excellent oxidation resistance, good mechanical behaviour at room temperature but creep even at moderate temperatures. As a result, carbon and SiC-based fibers presently are the most commonly used reinforcements in CMCs with a view to applications at high temperatures (say 1200–1800 °C). Small diameter ceramic fibers are extremely fragile and should be properly embedded in a refractory *ceramic matrix* (either oxide or non-oxide), primarily to protect the fibers and to permit load transfer from the matrix to the fibers. Fibers, with a volume fraction of the order of 40–50 %, are still the costly (but key) constituent of CMCs. *Nanoreinforcements*, such as carbon nanotubes or SiC nanofibers, are not presently used in CMCs to a significant extent, owing to handling difficulty, health considerations and cost.

Another key feature of CMCs, compared to polymer or metal matrix composites, is the fact that they are *inverse composites*, which is to say that under load it is the brittle matrix which fails first (in terms of failure strains: $\varepsilon_m^R < \varepsilon_f^R$) and at very low strain, typically ≈ 0.1 %. Hence, matrix cracks should be arrested or/and deflected at the fiber–matrix (FM) interface to avoid the early failure of the fibers and thus a brittle failure of the composite. This is achieved through a weakening of the FM-bonding, usually by introducing a thin layer (typically, 50 to 200 nm) of a weak material at the fiber surface, acting as a *mechanical fuse* and referred to as the *interphase*. The most commonly used interphase materials are those with a layered crystal structure (the layers being roughly parallel to the fiber surface and weakly bonded to one another to promote crack deflection). Examples are pyrocarbon (PyC) or hexagonal boron nitride (BN). Historically, they have been formed either (1)insitu in SiC (Nicalon)/glass-ceramic composites by decomposition of the fibers or/and FM interactions during composite high temperature (HT) processing, or (2) deposited by chemical vapour infiltration (CVI) from gaseous precursors, in C/SiC and SiC (Nicalon)/SiC composites. Both these processes were developed in the 1980s. When the FM bonding is weak enough, CMCs behave as *elastic damageable non-linear materials*, which is to say that beyond the proportional limit, the brittle matrix undergoes multiple microcracking under load, the cracks being deflected within (or near) the interphases, the fibers being partly (or totally) debonded and exposed to the atmosphere before the ultimate failure which commonly occurs at a strain of the order of 0.5 to 1.5 %. All these damaging phenomena take place with energy absorption and are responsible for the high toughness of the materials (a very uncommon feature for ceramics). An important milestone in this field was the pioneering work of A. Kelly and his coworkers in the early 1970s.

Improving the *oxidation resistance* of non-oxide CMCs is another important issue, particularly with a view to long duration exposures at high temperatures. In C/SiC and SiC/SiC composites, the weak points are the fibers themselves and the interphase. A first strategy is to use well-crystallized pure SiC fibers, which display good oxidation resistance, rather than SiC-based fibers (which usually contain free carbon and are poorly crystallized) or carbon fibers. In this field, the development of the so-called *stoichiometric* SiC fibers in Japan in the late 1990s was an important step. A second approach is to replace the commonly used PyC-interphase (which undergoes oxidation at temperatures as low as ≈ 500 °C) by *BN-interphase* (which displays a better oxidation resistance, at least in dry oxidizing atmospheres). A third possibility is to use *self-healing* coatings (single layer or multilayer), such as a SiC-coating containing a layer of a boron-bearing compound (such as B_4C) or a ternary Si–B–C mixture. The first role of the coating is to close the open residual porosity of the composite to impede the in-depth diffusion of oxygen. SiC-based coatings do undergo microcracking under load but oxygen diffusing along the microcracks would react with the microcrack wall to form a SiO_2–B_2O_3 healing phase. Finally, the best oxidation protection of SiC-based composites is achieved by extending the concept of crack-healing to the matrix itself which is now a *multilayered matrix* with layers of SiC, layers of sealant formers and layers of mechanical fuses, resulting in a lifetime under load exceeding 1000 hours at 1000–1200 °C. Finally, the use of oxidation-prone interphase can be avoided by utilizing a porous (and hence, relatively weak) matrix, but this approach exposes the fibers to the environment and, thus, it is better suited to oxide/oxide composites.

Processing considerations constitute another important point, the main requirement being that fiber degradation should be absolutely avoided. Hence, low temperature/pressure processing techniques are often favored. This is actually the case for the *CVI-process* and the *PIP-process* (Polymer Impregnation and Pyrolysis) where the matrix precursor is gaseous or liquid, respectively. Both are pressureless techniques involving temperatures of the order of 900–1200 °C. Further, the starting material can be a multidirectional fiber preform, e.g. a 3D-fiber architecture. These techniques yield near net shape parts (which can be of large size and complex shape) but with a relatively high residual porosity (10–15 %). In this field, an important milestone was the transfer of the CVI-process to the plant level in the 1980s for the volume production of C/SiC and SiC/SiC composites. In the so-called *RMI-process* (Reactive Melt Infiltration), the matrix is formed in situ by chemical reaction between a liquid precursor and a preconsolidated fiber preform. For SiC-matrix composites, the matrix precursor is liquid silicon (or a silicon alloy) and the fiber preform is consolidated (e.g. by PIP) with carbon, the former reacting with the latter to form the SiC-matrix. RMI is also a pressureless technique (it is conducted under vacuum). It yields near net shape composites with a low residual porosity, but it involves relatively high temperatures (1400–1600 °C for liquid silicon) with a risk of fiber degradation (unless thick fiber coatings are used), and the matrix usually contains unreacted precursor (such as free silicon). Finally, CMCs can also be fabricated according to *ceramic processing routes*. In the so-called Slurry Infiltration/High Pressure Sintering technique (SI-HPS), the reinforcement is impregnated with a suspension of matrix powder (usually a sol for oxide/oxide or a slurry for non-oxide matrix composites). After drying, the material is densified by sintering at high pressure. For non-oxide covalent ceramic powders, such as SiC-powders that display a poor sintering ability, sintering aids (such as oxide mixture forming eutectics) should be added to the slurry, the sintering conditions ($T = 1800\,°\mathrm{C}$, $P = 10–50\,\mathrm{MPa}$ for SiC) remaining harsh. As a result, only very stable fibers, such as the stoichiometric SiC fibers prepared at high temperatures can be used. This technique yields composites with almost no residual porosity, high crystallinity and high thermal stability, but it is not suited to the volume production of large parts with complex shapes.

CMCs are expected to have a serious impact on the development of new technologies, as suggested by a few successful current applications. Significant *weight saving* is achieved when heavy superalloys are replaced by high strength and tough C/SiC or SiC/SiC composites in aerojet or rocket engines. Fighters are already equipped with CMC engine nozzles and could have, in the future, CMC combustion chambers. *Lifetimes* of parts working at high temperatures are improved by replacing metal alloys by CMCs. A good example is given by CMC braking systems (C/C for aircraft and C/C–SiC for cars) which exhibit longer lifetimes than their steel counterparts and better wear and friction properties at high temperatures. The use of C/C brakes, first on military fighters and then on civil jumbojet aircraft, on the basis of weight saving, braking performance and safety considerations, as well as that of C/C–SiC brakes on Formula 1 racing cars and sport cars, constitute other important milestones. CMCs can considerably extend the *temperature domain* of use of structural ceramics in many fields, such as jet engines and gas turbines (with higher yields and the possibility of reducing (or even suppressing) cooling requirement), heat exchangers and high temperature chemical engineering. Another promising new field of application could be the use of SiC/SiC composites in high temperature *nuclear reactors* (fission and fusion) for power generation, on the basis of their refractoriness, high temperature mechanical properties (creep resistance), compatibility with neutrons and low residual radioactivity after prolonged exposure to radiation.

It thus appears that CMCs – compared to metal alloys and monolithic structural ceramics – constitute a new class of materials which are well suited to applications in harsh environments. However, they are still very new and will undoubtedly require an intense effort of research. Present applications in different demanding fields suggest that they could have a bright future in the development of high technologies.

Honorary Professor, Bordeaux 1 University *R. Naslain*

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Preface

Ceramic Matrix Composites (CMCs) represent a relatively new class of quasiductile ceramic materials. They are characterized by carbon or ceramic fibers embedded in ceramic matrices (oxide or non-oxide) with comparatively low bonding forces between the fibers and the matrix. These weak intefaces, in combination with a porous and/or microcracked matrix, result in composite materials which differ from all other structural materials or composites and show some outstanding properties. Their strain-to-failure is up to one order of magnitude higher than in monolithic ceramics and their low densities result in mass-specific properties which are unsurpassed by any other structural material beyond 1000 °C.

From their research beginnings about 40 years ago, the demands of space technology played the decisive role in the development of CMCs. Hot structures of limited lifetime (e.g. thermal protection systems, nozzles) in aerospace and military applications have been developed in different countries. In recent years, civil and terrestrial requirements became the driving forces and properties and manufacturing processes were consistently improved to transfer CMCs from niche applications to broader markets. Due to their high thermal stability and good corrosion and wear resistance, these composite materials are of increasing interest for long-term applications and damage-tolerant structures in different industrial sectors like ground transportation (e.g. brake and clutch systems), mechanical engineering (e.g. bearings, ballistic protections), and power generation (e.g. burners, heat exchangers).

The goals of further research and development are focused on improvements in the thermal and oxidative stability of the reinforcing fibers and on a considerable reduction of the processing costs. Reasonable costs for series productions are expected by using innovative continuously operated furnaces as they already exist for other structural (monolithic) ceramics. Also, new forming processes for the manufacture of green bodies and new hybrid processes of high reliability are necessary. Beside these fabrication approaches, novel precursors for cheaper ceramic fibers and improvements in the thermomechanical properties of short-fiber reinforced CMCs are key factors to develop CMC materials for wider application.

This textbook provides a comprehensive overview of the current status of research and development on CMCs. It presents data tables, process descriptions, and field reports, giving special emphasis to applications relevant to the respective topics. In this regard, the textbook begins with two chapters on fibers and textile preforms for the reinforcement of ceramic matrix composites, followed by the description of the fiber/matrix interfacial domain of CMCs. In this chapter, data on interfacial characteristics and techniques to measure these characteristics are provided. This is followed by four chapters describing the most important processes used to manufacture non-oxide CMC materials currently. This includes the manufacture of carbon/carbon, the melt infiltration of silicon into carbon/carbon composites, as well as the Polymer Infiltration and Pyrolysis (PIP) and Chemical Vapor Infiltration (CVI) processes. Two chapters on oxide-CMCs with dense and porous matrices, which are promising materials particularly in combustion environments, conclude the processing part of the book.

The following two chapters describe the microstructural modelling and testing of CMCs using different models and methods. These topics are of special interest for designing structural parts and predicting their lifetime, for example by integrating non-destructive testing methods. As all fabrication approaches have certain limitations in terms of size and shape, the following two chapters deal with machining and joining techniques to achieve CMC structures of high integrity. This is followed by chapters providing practical experiences of the application of CMC materials under extreme thermal as well as corrosive conditions. Hot structures in spacecraft and aircraft show the tremendous progress which has been achieved with respect to re-usability and lifetime of CMC structures over the last 20 years. The current stage of development in using SiC/SiC composites as future structural materials in nuclear applications is described in a separate chapter. The most attractive volume market for CMCs currently is the topic of the last chapter. Test results and experiences with high-performance brake and clutch systems equipped with disks and pads of C/SiC composites are presented, demonstrating their superior tribological behaviour in automotive and other applications.

I would like to thank all the authors for their valuable and timely contributions. I am grateful to Roger Naslain, one of the pioneers of ceramic matrix composites, for writing the Foreword of this book. Furthermore, I would like to thank Waltraud Wüst and her team from Wiley-VCH and Petra Jelitschek as well as Angelika Schwarz from my research teams in Bayreuth for their help and cooperation during the publication process.

Bayreuth, Germany Walter Krenkel

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1

Fibers for Ceramic Matrix Composites

Bernd Clauß

1.1

Introduction

New materials and processing routes provide opportunities for the production of advanced high performance structures for different applications. Ceramic matrix composites (CMCs) are one of these promising materials. By combining different ceramic matrix materials with special suitable fibers, new properties can be created and tailored for interesting technical fields.

This chapter gives an overview on fiber types, which can be used as fibrous components in CMCs [1–5]. The production of these fibers, as well as their structure and properties, will be discussed.

1.2

Fibers as Reinforcement in Ceramics

In CMCs, only fiber components are used that withstand the relatively high temperatures required for the production of ceramics, without significant damage. Other requirements to be met are long-term high-temperature stability, creep resistance, and oxidation stability. The importance of each of these demands depends on the type of application.

Organic, polymeric fiber materials cannot be used in CMCs because of their degradation at temperatures below 500 °C. Also conventional glass fibers, with melting or softening points below 700 °C, cannot be used for this purpose.

Possible candidates for the reinforcement of ceramic materials are polycrystalline or amorphous inorganic fibers or carbon fibers. The term “ceramic fibers” summarizes all non-metallic inorganic fibers (oxide or non-oxide), with the exception of fibers manufactured via solidification of glass melts.

The distinction between ceramic fibers and glass fibers has become more difficult during the last few years, because ceramics produced via new precursor or sol-gel routes can also be amorphous (i.e. “glassy”) in structure and the production process can also contain a melt processing step. This means that ceramic fibers can be

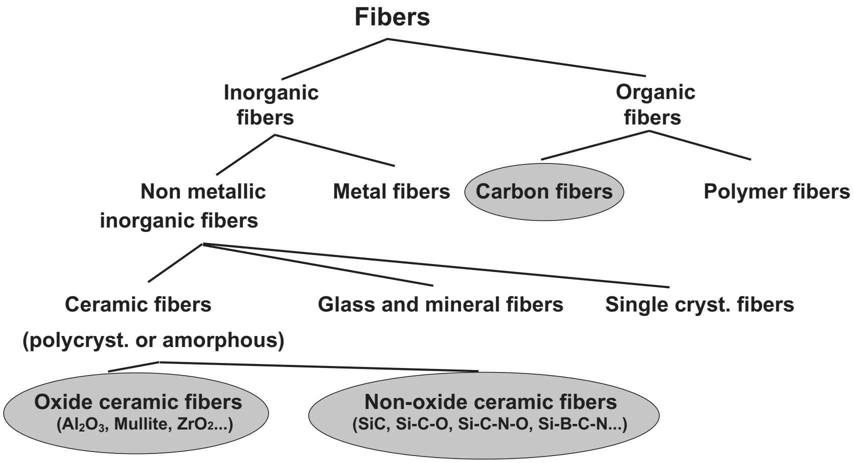


Figure 1.1 Classification of different fiber types.

either polycrystalline, partially crystalline, or amorphous. However, the expression “glass fibers” should only be applied to fibers that are produced via solidification of typical glass melts based on silicate systems. If these melts are produced by using minerals such as basalt, then the fibers should be called “mineral fibers.”

Carbon fibers can also be used under certain conditions in CMCs. Although these fibers degrade in an oxidizing atmosphere above 450 °C, they are stable under non-oxidizing conditions up to temperatures of 2800 °C. Carbon fibers have a very good cost-performance ratio, if an environmental protection of the composite allows the use of this fiber type. Therefore, environmental barrier coatings (EBC) is an important field of research in CMCs.

Figure 1.1 shows a general classification of fibers, containing ceramic fibers and carbon fibers.

1.3
Structure and Properties of Fibers

Fibers used in high-performance composites possess superior mechanical properties (and in CMCs also superior thermal properties) compared to “normal” polymeric fibers. This chapter will discuss how this can be achieved with fibers made from different materials, including polymers, glass, ceramics, and carbon.

1.3.1
Fiber Structure

As shown in Figure 1.2, the structure of fibers can be considered from different viewpoints, depending on the “magnification” at which the structure is presented. The example shown is a polymeric fiber.

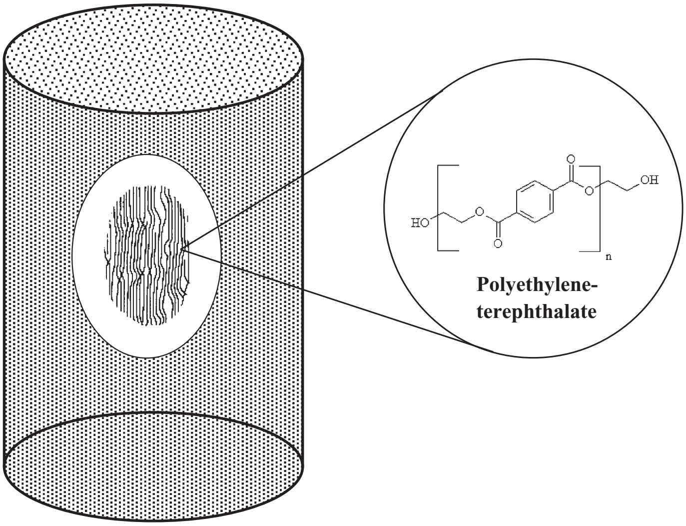


Figure 1.2 Fiber structure (example PET fiber).

At the *molecular level*, the displayed fiber is composed of poly(ethyleneterephthalate) (PET) chain molecules. This “chemical structure” determines the thermal and chemical stability and the theoretical strength of the fiber.

Most fibers also possess *supramolecular structures*, which are determined by molecular orientation and crystalline and/or amorphous regions (e.g. a two-phase crystalline and amorphous structure is indicated in Figure 1.2). These structures are formed during processing and can be significantly influenced by the processing conditions. This “physical structure” has important influence on the thermomechanical properties of fibers.

Finally, the fiber morphology influences the properties at the *macroscopic level*. Important criteria are cross-section, uniformity of the diameter along the fiber, porosity, and structural flaws, as well as surface properties such as roughness and surface energy (which is determined by the chemical structure of the surface). Adhesion to the matrix material and wetting behavior during infiltration steps are strongly influenced by these properties.

1.3.2
Structure Formation

The structure formation in fibers depends not only on the fiber material itself but also on the processing conditions. Process parameters are controlled in such a way that specific supramolecular and macroscopic structures can be obtained.

Important manufacturing processes are meltspinning, dryspinning, and wetspinning, together with modified spinning processes related to these:

- In the melt spinning process, fibers are formed via melts, which are forced through nozzles at high pressure and then solidified by cooling.
- In the dry spinning process, polymer solutions are used, which are also spun through nozzles. In this case, the fiber formation occurs by evaporation of the solvent from the spun solution.
- In the wet spinning process, polymer solutions are also used, but fiber forms by precipitation of the polymer in a liquid precipitation bath.

Important process parameters that influence the structure formation of the fibers are spinning speed, draw ratio, temperature, and other ambient conditions.

If specialty fibers are desired, the spinning process is often followed by after treatments, which will determine the final structure of the fibers.

Such after treatments include cross-linking of melt spun ceramic precursors before pyrolysis, annealing, and sintering of oxide based green fibers in order to achieve ceramic fibers and special surface treatments of carbon fibers.

If ceramic or carbon fibers are to be used in CMCs, the fibers have to be coated in many cases (e.g. with pyrocarbon or boron nitride), which act as interfaces between fiber and matrix.

1.3.3
Structure Parameters and Fiber Properties

The physical properties of fibers are determined essentially by three structure parameters: bond type, crystallinity, and molecular orientation [6].

The energy content of different types of chemical bonds is illustrated in Table 1.1. Covalent and ionic bonds, which can be oriented one-, two-, or three-dimensionally within fibers, have the highest energy content and so determine the mechanical strength and modulus of the fibers. Other bond types are of minor importance in high-performance fibers.

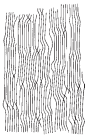
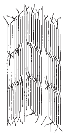
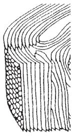
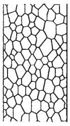
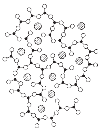
Different fiber types are shown in Table 1.2, which are distinguished by the structural parameters listed. It is obvious that the fibers possess different architectures and the final properties of the fibers (e.g. modulus and strength) are determined by a combination of these structural parameters.

If the bond type and the spatial orientation of the bonds were the main criterion for good mechanical properties, then ceramic fibers and glass fibers with three-dimensional, covalent, or ionic bonds would have far superior properties compared to other fiber types. But since these fibers are isotropic without molecular orientation, they possess lower strength than carbon fibers. This is because carbon fibers have a structure with two-dimensional covalent bonds, showing pronounced crystallinity and high orientation. Based on this advantageous combination of structural parameters, carbon fibers currently display the highest values for strength and modulus. Depending on the processing conditions, carbon fibers can achieve either moduli as high as 600 GPa, or extremely high strength values (above 7000 GPa).

Table 1.1 Chemical bond types and bond energies with examples of materials [6].

Bond type	Energy (kJ mol^{-1})	Examples	
Ionic	800–15 000	NaCl	3D
		ZrO_2	3D
		Al_2O_3	3D
Covalent	200–600 (single bonds)	Aramide	1D
		Graphite (in plane)	2D
		Glass, SiC	3D
Metallic	100–800	Metals	
Hydrogen bonds	20–50	Aramide	
		Aliphat. polyamide	
		Cellulose	
Dipole-dipole	ca. 2	Polyester	
van der Waals	ca. 1	Polyolefines	
		Graphite (between planes)	

Table 1.2 Fiber structures and properties [6].

Fiber type	Polyester polyamide	Aramide fibers from LC phase	Carbon	Ceramic (crystalline)	Ceramic (amorphous) glass
Structure	 1D linear 2 phases	 1D linear 1 phase	 2D layered	 3D isotropic	 3D isotropic
Bond type	1D covalent, hydrogen bonds (PA), dipole-dipole (PES), van der Waals	1D covalent, hydrogen bonds, van der Waals	2D covalent, van der Waals	3D covalent/ ionic	3D covalent/ ionic
Crystallinity	Medium	Paracyrstalline	Paracrystalline	Polycrystalline	Amorphous
Orientation	Medium	Very high	High	None	None

Polymeric aramide fibers also show high strength values due to their high molecular orientation in the fiber axis, although only one-dimensional covalent bonds and hydrogen bonds are present.

Figures 1.3 and 1.4 show an overview of mechanical properties of different fiber types. Averages are given because, as mentioned before, properties can differ depending on processing conditions. In reality, no perfect fiber structure can be

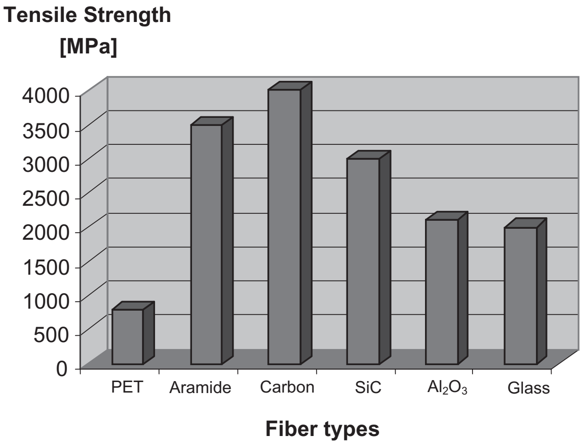


Figure 1.3 Typical tensile strengths (averages) of different fiber types.

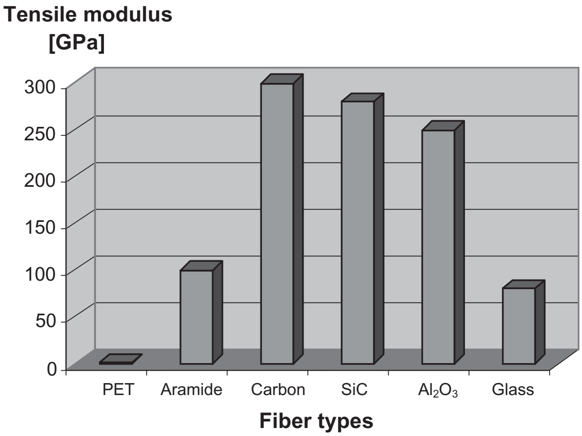


Figure 1.4 Typical tensile moduli (averages) of different fiber types.

obtained during processing, which means that the real properties of fibers are usually far below the theoretical property values calculated for a perfect structure. Therefore, one important goal of a fiber spinning and fiber formation process is to reduce structure imperfections to a minimum by optimized process control. Relatively high prices of high-performance fibers are usually caused by highly sophisticated and complex manufacturing processes rather than by more expensive materials used in the fiber production.