

Essential Properties of Fibres for Composite Applications

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Abstract In this chapter, essential properties of fibres required for fabricating good performance composites are presented. Fundamental aspects of these properties and principles of their characterization techniques are discussed. Firstly, the geometrical aspects of fibres (for short and endless fibres) are described. The second part deals with the different types of structures in fibres with respect to molecular orientation mostly responsible for anisotropy of fibres. In the third part, the mechanical properties (especially tensile properties) and failure mechanisms for different types of fibres are discussed and correlated to the structure of the fibres. The following part is concerned with the surface of fibres, which is responsible for the interaction of the fibres with the matrix material in composites and has a large influence on the wetting behavior and adhesion to matrix materials. In the last parts, further physical properties (heat capacity, thermal conductivity, thermomechanical properties and electrical conductivity) and the durability of fibres are described.

1 Introduction

Fibers are a unique class of materials because of their anisotropy. Usually, fibers have a high length compared to their diameter. This so called high aspect ratio is responsible for the unique properties compared to a bulk material. Beside the geometrical aspect of anisotropy, the most important fiber types also have an anisotropic structure. Both aspects together result in anisotropic physical properties, which are described in this book chapter.

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First of all, the geometric and structural aspects of anisotropy result in extraordinary mechanical properties. Typical fibers for composite applications have high strength and stiffness in fiber direction, while they are weak and flexible perpendicular to it. Also other physical properties, like electrical or thermal conductivity may be totally different when measured along or perpendicular to a fiber. When embedded into a matrix, these anisotropic properties can also be transferred to the fiber reinforced composite.

In this case, the mechanical and other physical properties can be tailored in the composite part depending on the placement of fibers. For designing composites with tailored properties, it is essential to understand the anisotropic properties of fibrous materials.

Another unique property is the high surface area of fibers, caused by their small diameter compared to their length. The fiber surface with its topography and chemistry is the interface to the matrix material. First of all, the fiber surface determines the wetting behavior with matrix material, where surface tension of the materials and capillary effects have to be taken into account. Secondly, the interface between fiber and matrix is responsible for the transfer of load to the fiber. By controlling the adhesion between the two components, the failure of a composite part can be adjusted to specific needs. A detailed understanding of the interaction of fiber and matrix is therefore necessary to design tailored fiber reinforced composites.

The geometrical properties, the fiber structure, the most important physical properties of fibers and the fiber's surface are described in this chapter with special respect to the application of these materials in fiber reinforced composites. Furthermore, suitable methods for analyzing these properties are presented. These fundamentals will help the reader of this book to understand the specific properties of fibers and composites in the following chapters.

2 Geometrical Properties

The geometrical aspects of fibers are mainly defined by their length L and diameter D , resulting in the so called aspect ratio L/D . Concerning the fiber's diameter, different shapes of fibers beside a round shape are possible. Furthermore, the so called fineness or titer T , which is the mass per length unit, is an important factor to describe the fibers geometry, since it takes into account differences in density of the specific fiber types [1].

2.1 Cross-Section

The most common synthetic reinforcement fibers have a round cross-section, since it provides a uniform mechanical loading of the fiber material. This leads to a higher specific strength of the materials compared to other fiber shapes [1, 2].

However, different cross-sections can be found in fibrous materials for composite applications

- (1) *Process-based deviations from round cross-section:* Depending on the spinning process of the reinforcement fibers, non-round filaments can be formed due to inhomogeneous processing conditions. A typical examples are carbon fibers with the so-called “kidney-shaped” cross-section. This cross-section is formed in the wet-spinning process depending on the coagulation conditions (see Fig. 1) [3, 4].
- (2) *Intended deviations from round cross-section:* By changing the shape of the spinneret during fiber production or specific post-treatment of the fibers, different fiber cross-sections can be achieved. Examples are star-shaped or triangular fibers [5]. Furthermore, hollow fibers are common for creating lighter materials or materials with special transport properties (e.g. in membranes) [6]. Beside the mechanical properties of the filaments, rovings and composites, the fiber cross-section can have an influence on the processing of the fibers (e.g. tribology or wetting behavior).

Beside the outer (geometrical) shape of the fiber, the material distribution within a filament can differ from a homogeneous distribution. This deviation can also be caused by the process (e.g. core-sheath structures in wet spinning or carbon fiber conversion) or be intended (e.g. bicomponent fibers made of two polymers) [3, 7].

The (geometrical) cross-section and the material distribution is usually analyzed by light or electron microscopy, depending on the necessary spatial resolution [8]. By combining microscopy with spectroscopic methods (e.g. Energy-dispersive X-ray spectroscopy), information about the chemical composition over the cross-section can be obtained.

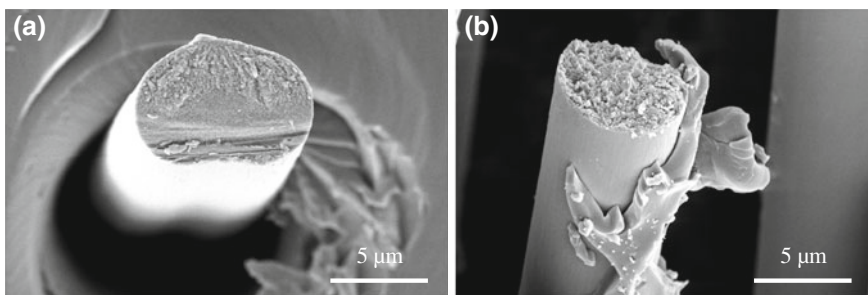


Fig. 1 Scanning electron microscopy (SEM) pictures of **a** “kidney-shaped”, **b** round carbon fibers

Table 1 Classification of fiber length for textiles and plastics processing

Fiber type	Length's range (textiles) (mm) [1]	Length's range (plastics processing) (mm) [9]
Infinite length	>200.0	>50.0
Long staple fibers	40.0–200.0	1.0–50.0
Short staple fibers	6.0–40.0	
Short fibers/pulp	<6	0.1–1.0

2.2 Fiber Length

For the fiber's length, short or staple fibers ($L/D < \infty$) and fibers with infinite length ($L/D = \infty$) have to be taken into account [9, 10]. In the following Table 1, the classification of fibers length for textiles and plastics processing is displayed.

The length and its distribution of fibers with finite length is usually analyzed by microscopy [11]. Because of the small number of fibers analyzed by one microscopic image, novel methods for analyzing length and diameter of a large number of fibers were developed.

2.3 Fiber Fineness

The fineness (or titer T) of a fiber describes the mass per unit length. Therefore, it depends on the diameter d of the fiber and its density ρ (see section below). The unit for fiber fineness is tex (grams per 1,000 m) or den (grams per 9,000 m). The following equation can be used to calculate the fineness from filament diameter or vice versa [1]:

$$D = \sqrt{\frac{4}{\pi \cdot \rho} \cdot T} \tag{1}$$

The fineness can be measured either for complete rovings or for single filaments. For rovings, a reel is used to prepare fibers of a fixed length (usually 100 m) for the determination of their mass. The fineness of single filaments can be determined by analyzing their resonance frequency f at a defined length l and pre-tensioning with a force F . The following equation allows the calculation of the corresponding fineness [12]:

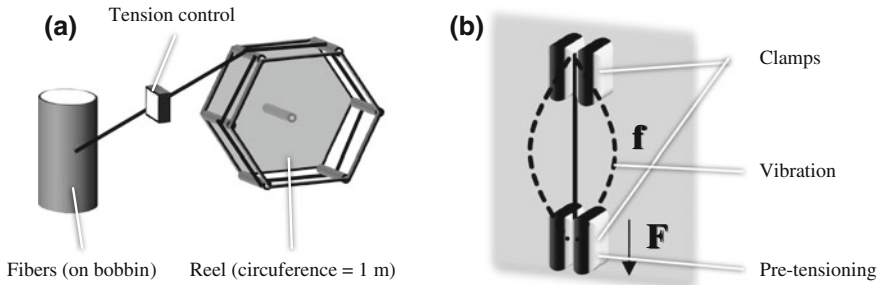


Fig. 2 Illustration of methods for determining the fineness of fibers (according to [12]). **a** Determination of weight per length by reeling. **b** Measuring single filament diameter by analyzing the resonance frequency during vibration

$$T = \frac{F}{4 \cdot f^2 \cdot l^2} \quad (2)$$

The methods for the determination of fiber fineness are displayed in Fig. 2.

3 Fiber Structure

The structure of a fibre is responsible for all of the properties described in this chapter. Therefore, a basic overview on the structures present in a fibre shall be given. The structure of the common reinforcement fibres can be considered as hierarchical, in which a certain structural element is used to build the next larger part of structure, so that the size of structures in a single filament range over several orders of magnitude (from 10^{-10} m, the distance between two atoms, to 10^{-5} m, the diameter of a filament). Due to the anisotropy of fibres, special attention has to be paid also to the anisotropy of structure. Several structural elements can be oriented in a filament, depending on the material [13–15]. The different types of structures are illustrated in Fig. 3 and described in the following sections.

3.1 Description of Structures

The *atomic or molecular structure* is being formed by the interaction of single atoms and the resulting type of chemical bonding. Depending on the type of chemical bonding, anisotropic structures can be formed on the molecular level. Usually, covalent bonding between atoms causes such an anisotropy (such as graphene layers in carbon fibres and the backbone in polymeric fibres), whereas metallic or ionic bonds are isotropic.

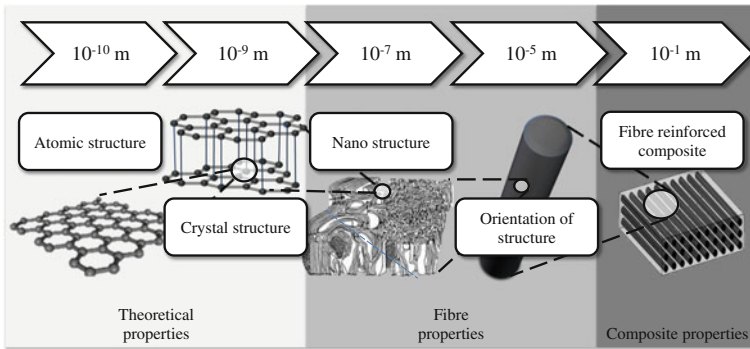


Fig. 3 Types of structures in a reinforcement fibre with their size indicated (structure illustrated exemplarily for carbon fibres) [16]

Depending on a regular or non-regular atomic or molecular structure, the atoms respectively molecules can be arranged to a *crystalline structure* or non-crystalline (amorphous) structure. In crystalline structures, atoms or molecules have fixed distances to each other in all space directions over a long distance, which are described by the unit cell. Amorphous structure are contrarily characterized by a medium (most probable) distance to the next atom or molecule. However, also the size of crystalline structures is limited and crystallites of different sizes and shapes can be formed in a fibre. If the molecular structure is anisotropic, usually anisotropic crystalline structures are formed, which may have an anisotropic (needle, disk- or platelet-like) shape. The crystalline or amorphous structure of a material usually defines the theoretical properties of a material [13, 14, 17].

Different crystallites or amorphous structures are combined to the *nano structure* of a fibre. It is characterized by the arrangement of the single elements, which may differ over the diameter of a filament [18]. Depending on the molecular architecture, crystalline and amorphous structures as well as nano-sized pores coexist in the nano-structure of a filament [19]. The nano structure is usually anisotropic, and can even be anisotropic if the single elements are isotropic or amorphous, such as in glass fibres.

The different elements of the nano structure as well as the nano structure itself may have a distinguished *orientation* in the filament. Usually, anisotropic crystalline structures such as in polymeric or carbon fibres can be highly oriented in the direction of the fibre [3, 18, 20]. The assembly of the single elements to the nano structure as well as their orientation define the properties of the material in the fibrous form.

The most outer parts of the nano structure form the *surface* of a filament. Therefore, the arrangement and orientation of structures in the outer parts of a filament can have an effect on the surface morphology (and roughness) as well as the possibilities of adding chemical functionalities to the surface. This part of the structure is then responsible for the interaction with the matrix material and has an influence on the composite properties such as wetting behavior and adhesion.

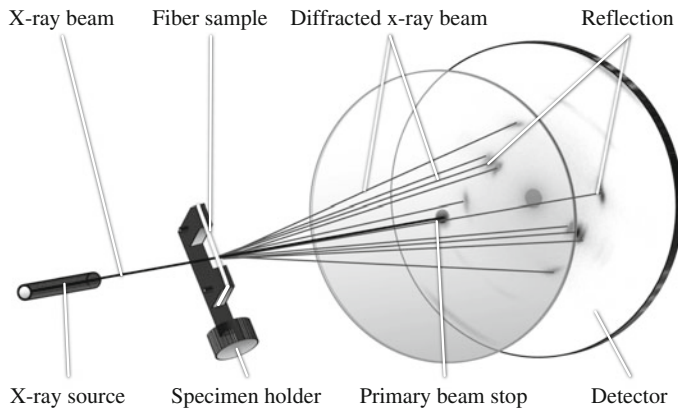


Fig. 4 Illustration of WAXS setup for analyzing structure and orientation of fiber samples

3.2 Structure Analysis by X-Ray Diffraction

The structural features of a fiber are usually analyzed by diffraction and scattering methods. The most important method for structure analysis is the diffraction or scattering of X-rays, while Wide Angle X-ray Diffraction (WAXD, or Wide Angle X-ray Scattering—WAXS) is used for the evaluation of the atomic and crystalline structure of a material. A typical setup for analyzing fibers by WAXS is displayed in Fig. 4. By using two dimensional detectors, both the structural features as well as their orientation within the fiber can be analyzed [15].

Most of the primary X-ray beam with the wavelength λ is transmitted through the fiber sample. However, the X-rays are partly scattered elastically. Depending on the distance of scattering structures and their orientation in the fiber, positive interference takes place at certain angles to the primary beam (diffraction angle 2θ) and relatively to the fiber axis (azimuthal angle ϕ) [21].

Structural features of a fiber are analyzed by the evaluation of intensity distribution as a function of the diffraction angle 2θ . As indicated in Fig. 5, the intensity has to be integrated first over the azimuthal angle for taking into account all structural features independent from their orientation. The resulting intensity diagram can then be used to fit peaks to the data. Typically, sharp peaks from crystalline structures and/or broad peaks from amorphous structures can be found in the intensity distribution [15].

The center position of a peak is connected to the distance within the crystalline lattice by Bragg's equation, while d_{hkl} is the distance for arbitrary lattice planes [21]:

$$\lambda = 2 \cdot d_{hkl} \cdot \sin(\theta) \quad (3)$$

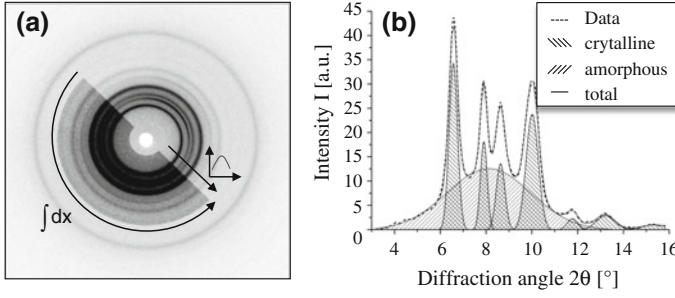


Fig. 5 Analysis of structural parameters from WAXS 2D images. **a** Integration of intensity over azimuthal angle. **b** Intensity as a function of diffraction angle for data fitting (crystalline and amorphous peaks)

The width of the peaks is determined by the finite size of the structures (or the size of domains within a structure, which are correlated to each other). The peak width $\Delta 2\theta$ can therefore be used for the determination of the expansion D_{hkl} of structures in the direction of an arbitrary lattice plane by using Scherrer's equation (K is a constant factor determined by the crystalline structure) [15, 21]:

$$D = \frac{K}{\Delta 2\theta \cdot \cos(\theta)} \quad (4)$$

The number of scattering structures determines the total intensity of X-ray beam which is scattered. If two or more different phases (e.g. crystalline and amorphous) are present in a fiber, the phase content X can be determined by determining the relation of the intensity I_x of a specific phase relatively to the overall intensity I_{total} scattered by the fiber [15]:

$$X = \frac{I_x}{I_{total}} \quad (5)$$

By analyzing the azimuthal distribution of intensity, the orientation of structures can be determined. The peak intensity in the direction of diffraction angle has to be integrated and evaluated as a function of azimuthal angle (as displayed in Fig. 6).

By analyzing the center position of peaks φ_c , the orientation of specific lattice planes relatively to the fiber axis is analyzed. The standard deviation $\langle \sin^2(\varphi - \varphi_c) \rangle$ from this position indicates the degree of orientation, which is expressed by Herrmann's orientation factor f for uniaxially oriented structures [14, 15]:

$$f = 1 - \frac{3}{2} \cdot \langle \sin^2(\varphi - \varphi_c) \rangle \quad (6)$$

Contrarily to WAXS, the method of Small Angle X-ray Scattering (SAXS) can be used to analyze the nanostructure of fibers. By analyzing intensity distribution at

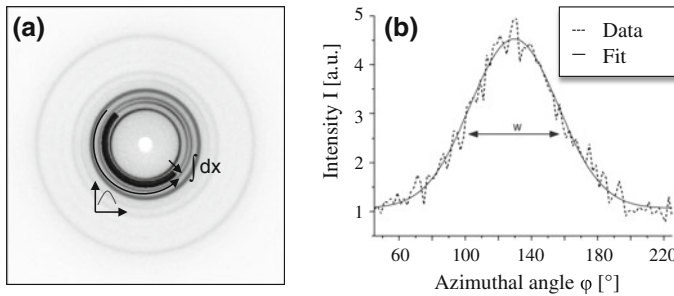


Fig. 6 Analysis of orientation parameters from WAXS 2D images. **a** Integration of intensity over diffraction angle. **b** Intensity as a function of azimuthal angle for data fitting (determination of width of crystalline peaks)

lower diffraction angles, scattering from objects with larger distances can be analyzed according to Bragg's equation. SAXS can be used to identify fibrils or other types of secondary structure formed in a fiber [14, 18].

3.3 Other Techniques for Structure Analysis

Beside X-rays, other types of radiation (e.g. neutrons or electrons) can be used to perform scattering experiments. Furthermore, other analysis techniques can be used to evaluate the structure of fibers:

- A direct visualization of structures is possible by microscopic methods such as electron microscopy. Especially with transmission electron microscopy (TEM), structures can be visualized with nearly atomic resolution. Because of high electron intensity necessary for that spatial resolution, only fibres with satisfying temperature stability and electrical conductivity (e.g. carbon fibres) can be analyzed in that way [22]. Furthermore, the area to be analyzed is very small (usually in a range of <100 nm), resulting in poor statistics of the analyzed data.
- Spectroscopic methods (such as IR, Raman or NMR spectroscopy) can be used to obtain information about the chemical bonds in a fiber, which allows the identification of the structures which are present. A quantitative description of structures as known from X-ray diffraction is however only possible if the method is calibrated with samples with well-known structural features [14].

4 Mechanical Properties

Typical reinforcement fibers have excellent tensile properties in fiber direction allowing high load transfer from the matrix in this direction, while they are relatively weak if loaded perpendicular to the fiber axis. This results in a high flexibility during processing, but also to fiber breakages during bending or if fibers are not well oriented. The relevant properties to describe tensile and bending behavior are described here.

4.1 Tensile Properties

The relevant measures to describe the tensile properties of reinforcement fibres are there tenacity, maximum elongation and Young's modulus [1, 3].

A high tenacity σ (or strength, stress at maximum elongation) is necessary for acting as a reinforcement of the matrix material. The strength of the fibres is usually one or two orders of magnitude higher than the strength of the matrix material. If mechanical load is transferred completely from the matrix to uniaxially oriented fibers (see section surface properties), the composite strength can reach the fiber strength multiplied by the fiber volume content. For reinforcement fibres, tenacity is usually measured in the unit of MPa or GPa. For lightweight construction, the specific tenacity (tenacity divided by the fiber's density) is indicated additionally to take into account the density of different material classes [3, 9].

The maximum elongation $\epsilon_{\max}$ of reinforcement fibres is usually low compared to the possible elongation of the matrix material. In case of good load transfer, composite failure takes place at the level of maximum fibre elongation due to fibre breakages. The failure behavior of the composite is usually brittle. By adjusting fiber matrix adhesion (see section surface properties), a more ductile failure behavior can be achieved if cracks propagate slowly between the single filaments [3, 9, 10].

Young's modulus E describes the stiffness of the fibre measured in the region of linear elastic deformation and is indicated usually in GPa. Fibres with high Young's modulus have a positive impact on the stiffness of the composite part if load is transferred sufficiently from the matrix [9].

4.2 Tensile Test Methods

Tensile properties of fibres can be measured on single filaments or complete rovings. For single filaments, standard test methods [23] and special testing machines (e.g. Favimat by Textechno GmbH, Mönchengladbach, Germany) can be used [24] (see Fig. 7a). For standard test methods, filaments are fixed on a paper frame, which is

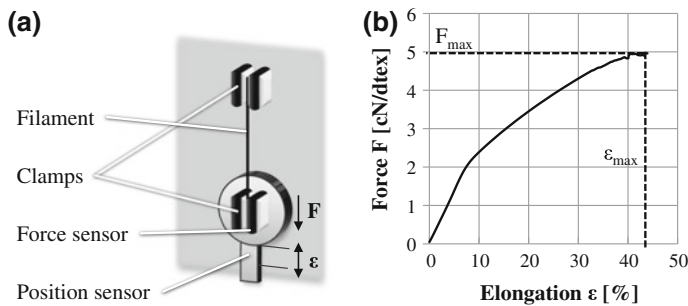


Fig. 7 **a** Experimental setup for single filament testing (Favimat method). **b** Resulting stress-strain diagram of a polymeric fiber

transferred to the testing machine and then cut before the tensile test. Stress-strain diagrams should at least be measured at 50 filaments [23]. A typical stress-strain diagram of a single filament is displayed in Fig. 7b. Maximum force (or tenacity) and maximum elongation can be easily extracted from the diagram. For the evaluation of Young's modulus, the linear elastic region (usually below 1 % of elongation) has to be taken into account [23].

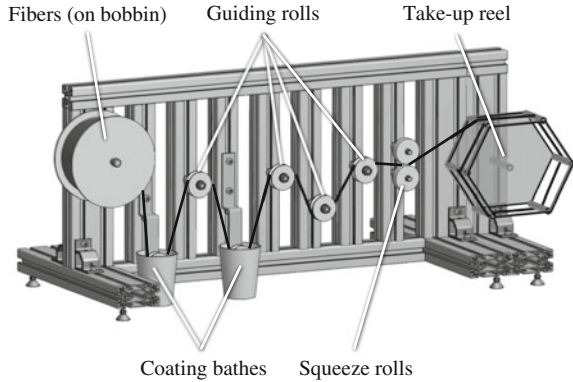
For tensile tests on rovings, the following test methods are available:

1. The ends of the fiber roving are placed between two clamps, which can be fed automatically (e.g. by special testing machines like Statimat from Textechno GmbH, Mönchengladbach, Germany). This method is suitable if a sufficient load transfer between all filaments and the clamps can be achieved and the filaments are not too brittle to break between the clamps. It is therefore usually applied for polymeric fibres with low fineness.
2. Fibre ends are impregnated with a suitable resin at a defined geometry, which fits into a standard testing machine. The method can be applied to avoid filament breakages at the clamps. However, it is only valid if the tenacity is not too high, since fibre pull-out can occur at the resin forms (see section surface properties).
3. The standard method for glass and carbon fibres requires a complete impregnation of the rovings with a suitable resin [25, 26]. Only by a complete impregnation, a complete load transfer to all filaments without fibre pull-out can be achieved. A valid setup for the coating of rovings is displayed in Fig. 8. The coated rovings can then be placed into a standard testing machine.

4.3 Effect of Fibre Length and Diameter

Both the fibre gage length in mechanical tests and the filament diameter have an influence on the measured strength of a fibre. Defects in the fibre structure are mainly responsible for their failure. The effect of length and diameter depend on the

Fig. 8 Experimental setup for coating glass or carbon rovings according to [25, 26]



probability to find a critical defect in the test volume, which is responsible for the failure of the fibre [2].

The probability to find a critical defect in a fibre of a length l is determined by the Weibull statistics. The resulting mean strength of $\langle \sigma \rangle$ is then a function of fibre length with $\ln \langle \sigma \rangle \sim -\ln(l)$ [27].

The relation for the fiber diameter is similar. Also here the probability of finding a defect within the fibre cross-section area A has to be taken into account. The impact of diameter on the measured mean fibre strength depends on the type of material used in the tests, e.g. $\langle \sigma \rangle \sim 1/d^{1/2}$ for glass fibres [2].

4.4 Bending Properties

While the bending properties of a composite can be easily determined by standard test methods, the flexural strength and bending stiffness of single fibres are only accessible by microscopic test methods at filament level.

Three-point bending tests can be implemented on the filament level by using very precise testing machines to measure the flexural strength (see Fig. 9a), since maximum forces in bending mode do not exceed the range of several mN. Fibres have to be fixed with a length of several 100 μm and bending has to be performed with a probe of a very small diameter, usually not exceeding 1 μm [28]. Another test method is based on applying a compression load in the fibre direction (see Fig. 9b). This leads to a bending deformation, so that flexural strength and bending modulus can be calculated [29].

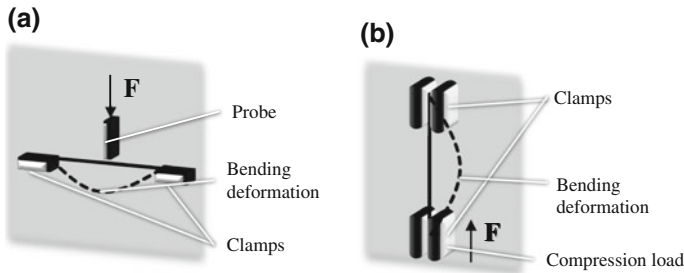


Fig. 9 Methods for measuring bending properties of filaments. **a** Three-point bending. **b** Application of compression load

5 Surface Properties

As the interface between fiber and matrix material dramatically influences a composite's performance, the fibers' surface properties are an important part of the reinforcing materials.

The parameter which characterizes the binding strength of fiber and matrix is called interlaminar shear strength (ILSS); its unit is $[N/mm^2]$ [3]. The value of the ILSS between fiber and matrix is determined by mainly by two influencing factors: the mechanical and the chemical interlock [30].

In order to modify the degree of adhesion between fiber and matrix, most reinforcement fibers are not directly embedded into the composite matrix. They will undergo a surface treatment (leading to chemical and structural changes on the surface) as well as a coating procedure called sizing application (the sizing has the double function to protect the fiber during textile processing and promote adhesion in the composite).

Apart from the resulting adhesion properties in the composite, also wettability of the fiber with matrix materials is important during impregnation.

In order to characterize the properties mentioned above, analytical methods for their determination are presented in the following section.

5.1 Surface Area and Surface Roughness

The most common synthetic reinforcement fibers have a diameter in the range of less than $50\ \mu m$, while pores and fibrils in the range of nm contribute to the surface which is later bound to the matrix material.

Apart from the adhesion properties, the surface area and roughness have an impact on the fibers' tribological properties [31]. These properties are important when it comes to fiber-fiber-interaction and friction with machinery elements during textile processing. If the surface is too rough and a protection (e.g. in form of a

sizing) is missing, a lot of fiber breakage and hence a weakening of the reinforcement fiber material will occur [32].

Therefore, analytics for determining the surface area of reinforcement fibers also has to be sensitive in a nanometer range. As a quantitative characterization method for the determination of the fiber's surface, BET measurements on fibers are presented in this section.

Apart from the quantitative measurement of a surface area, it is also important for composites to know the surface roughness of the fiber material as well as imaging the surface in order to know the different roughness values in different fiber directions.

5.1.1 BET Surface Area Measurements (Named After Stephen Brunauer, Paul Hugh Emmet and Edward Teller)

In this measurement method, the phenomenon of physical adsorption of gases on solid surfaces is used to determine the surface of the material analyzed.

For the measurement, the fibers are put into a sample holder (usually a cavity similar to a glass test tube) which is connected to a gas supply. From the cooled gas supply, the gases (nitrogen, argon or krypton at their boiling temperature) can float to the cavity with the samples (see Fig. 10a). The sample holder is cooled down to temperatures lower than the triplex point of the measurement gas, leading to a volumetric measurement with a precision of 0.5 % or better [33].

The pressure in the sample cavity is constantly held on the level of the equilibrium pressure p_0 , where as many adsorptions as desorptions take place (see Fig. 10b). When the pressure value goes below p_0 due to adsorption of the gas on the sample's surface, more gas is guided into the sample cavity. The amount of gas which floats into the cavity is characterized by the according pressure p . The relation p/p_0 is plotted against the number of particles adsorbed. By taking into

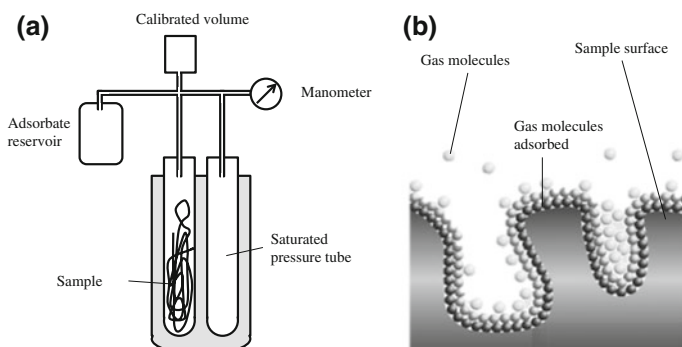


Fig. 10 **a** Measurement setup for BET surface area measurements (static volumetric method); **b** gas adsorption on a solid surface: the phenomenon used in BET surface area measurements

account the cross-section of one gas molecule adsorbed to the surface and the number of layers on the surface (BET theory [34]), the specific surface area per weight unit (m^2/g) is calculated.

5.1.2 Atomic Force Microscopy (AFM)

With atomic force microscopy (AFM), the topography of surfaces is characterized by measuring the inter-atomic forces between the sample surface and a measurement tip. The tip end ideally consists of only very few atoms. The tip is mounted on a cantilever which is oscillating with a frequency close to its resonance frequency. A piezo motor approaches the tip to the surface until the amplitude is lowered by the inter-atomic forces. The height information of the piezo element and the difference in the amplitude are recorded while the measurement process is repeated for every point of the sample to be measured [35]. The experimental setup is shown in Fig. 11a).

As the measurement is time-consuming, only parts of the surface can be characterized by this method. Typically, the size of AFM scans lies in the range of 2.5 or 5 μm^2 . The number of points to be measured per line are always powers of two (256, 512 or 1024) due to digitalization. With the data taken, height information is available all over the scan area. Therefore, roughness values in different directions can be analyzed. Moreover, a three-dimensional image of the surface area scanned can be calculated (see Fig. 11b).

5.2 Chemical Functionalities

In order to evaluate the possibilities for chemical interlock between fiber and matrix material it is necessary to analyze the types of functional groups on the surface. This

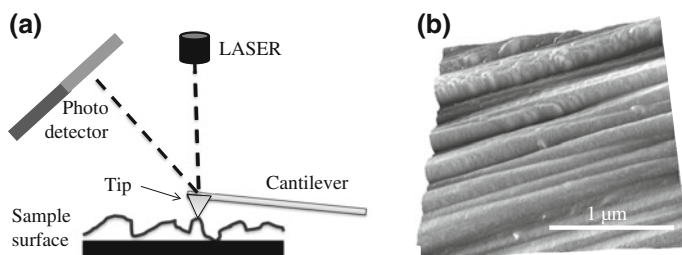


Fig. 11 **a** Measurement setup for AFM. **b** AFM image of a carbon fiber surface before surface treatment

can be done by different spectroscopic methods. Two spectroscopy methods often used for determining functional groups on fiber surfaces are presented here.

5.2.1 X-Ray Photoelectron Spectroscopy (XPS)

XPS is a method for the quantitative analysis of the elements on a solid surface. The measurement method the phenomenon that electrons are separated out of a solid by electromagnetic radiation. When the solid is exposed to X-ray source, electrons are hit by photons from the X-ray radiation with the energy $\hbar\omega$. If the energy transferred to the electron is sufficient to overcome the binding energy E_b and the electric potential Φ , the electrons will leave the solid with their rest energy E_{kin} . The energy balance equation is

$$\hbar \cdot \omega = \Phi + E_{kin} + E_b \quad (7)$$

As ω is known from the X-ray source applied, while the other factors are constants, the binding energy E_b can be calculated when the kinetic energy is measured. The binding energy E_b is characteristic for the atomic or molecular orbital where the electron comes from. Hence, the information on the binding energy of the separated electrons lead to the knowledge of elements on the specimen surface investigated [36].

The measurement setup for XPS measurements is shown in Fig. 12. The x-ray source provides the photons which excite the electrons on the sample's surface (depth of the electrons up to 3 nm). The electrons with sufficient kinetic energy will reach the hemisphere electron energy analyzer where the voltage applied between to concentric electrodes can select the electrons which will reach the detector. An electron multiplier transforms the weak electron signal into a measurable signal. The resulting spectrum is a graph with the counts per second versus the binding energies E_b . A Comparison of the binding energies with a database leads to the information of the elements and molecules on the surface [37].

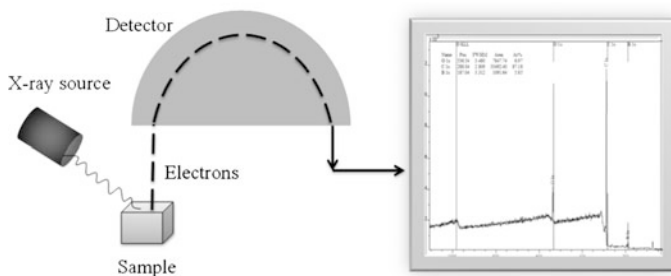
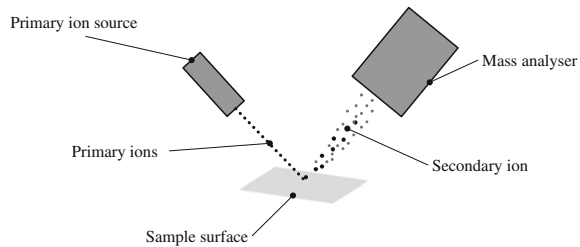


Fig. 12 Measurement setup for XPS

Fig. 13 Measurement setup for ToF-SIMS



5.2.2 ToF-SIMS (Time-of-Flight Secondary Ion Mass Spectroscopy)

In this measurement method, a sample's surface is exposed to ions (e.g. O_2^+ , Cs^+ , Ga^+ , Ar^+ , Bi^+) which are accelerated by a voltage of 0.2–30 keV. During the interaction of the accelerated ions, new ions (charge: neutral, negative, positive) are emitted. The measurement setup is set under a high vacuum (approx. 10^{-7} mbar). When the newly formed ions are leaving the sample's surface, they all have the same energy. Hence, light ions have a higher velocity than heavier ions. This velocity difference is used when a time-of-flight-analyzer is employed for the measurement: this analyzer consists of a vacuum tube with a fast detector at its end [38]. The experimental setup is displayed in Fig. 13.

The method is very sensitive and can separate ions as well as ion clusters up to $\frac{m}{\Delta m} > 10.000$. Therefore, also ion clusters with the same mass number can be separated. The measurement data for the ion masses are compared to a database. With the knowledge on the ions which were accelerated towards the surface (primary ions) and the comparison with the newly formed ions measured in the analyzer (secondary ions), an evaluation of the molecular composition of the sample's surface is possible [39].

5.3 Surface Energy and Wettability

When fiber materials shall reinforce matrix materials, the textile structures have to be impregnated with the matrix material still in a liquid condition. The processes for impregnating textiles with resins vary according to the matrix materials used and according to the application of the composite [10]. All processes have in common that the degree and quality of impregnation strongly influence the properties of the resulting composites. The wettability of the fiber material is crucial for the set-up of process parameters, as the wetting behavior has a strong impact on the cycle time for the impregnation process.

In this section, at first the impact of surface energy on the wettability are explained. Then, measurement methods for the wettability of fibers with matrix materials are explained.

5.3.1 Surface Energy and Contact Angle

As an indicator for the ability of a liquid to wet a material, the contact angle of a droplet of this liquid on a plain horizontal solid surface can be measured. The contact angle α is very small when the droplet immediately loses its form after contact with the surface. Hence, the wettability of the surface with the liquid is very high. If the wettability is not good, the contact angle α lies in the range between 180 and 90° [40] (see Fig. 14).

The wettability and hence the contact angle α depends on surface energies of the materials involved in the wetting process. The surface energy σ is defined as the amount of energy ΔW which has to be overcome in order to increase the surface by ΔA :

$$\sigma = \Delta W / \Delta A \quad (8)$$

The work ΔW which has to be overcome is due to the intermolecular cohesion within the liquid. If a liquid is brought on a surface, then the tension of the boundary layer σ_{LS} becomes important (see Fig. 15).

Following the equation $\sigma_L \cdot \cos \alpha = \sigma_s - \sigma_{LS}$, good wettability is achieved when the surface energy of the solid (fibers) is higher than the surface energy of the wetting liquid (e.g. matrix material).

Surface energies can be separated into a polar part σ_p (due to polar chemical bonds, e.g. hydrogen bridge linkage) and an unpolar part σ_u (due to non-polar chemical bonds, e.g. van-der-Waals forces) [41]:

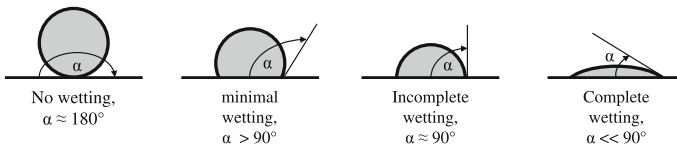


Fig. 14 Liquid droplets showing a different wetting behavior

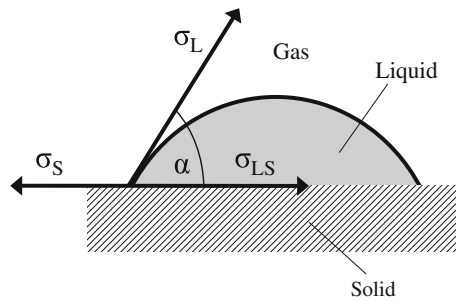


Fig. 15 Relations between surface energies of liquid and solid determine the contact angle and the resulting wettability

$$\sigma = \sigma_p + \sigma_u \quad (9)$$

5.3.2 Measurement of Surface Energies and Wetting Behavior

The surface energy of liquids can be measured by the Wilhelmy method, employing an inert testing body which is dipped into the liquid [40]. The measurement of the surface energy of fibres is performed by using testing liquids with a defined surface energy. Using testing liquids with defined polarity leads to discovering the polarity properties of the solid (fibres) [42].

These measurements are performed with a tensiometer (see Fig. 16a), which measures the weight increase of the specimens dipped into the testing liquid [43]. If single filaments (Fig. 16b) are measured, a sufficient number of samples has to be measured in order to provide statistical significance.

The wetting behavior of fiber bundles (Fig. 16c) is described by the Washburn equation [44], while the measurement is carried in a set-up developed for powder materials [45]. The method takes into account capillary effects (expressed with the capillary constant C) and properties of the testing liquid (viscosity η , density ρ_L and surface tension σ_L). With the following equation, the contact angle can be determined from the measurement of the weight increase per time:

$$\cos \theta = \frac{2 \cdot \eta}{C \cdot \rho_L^2 \cdot \sigma_L} \cdot \frac{m^2}{t} \quad (10)$$

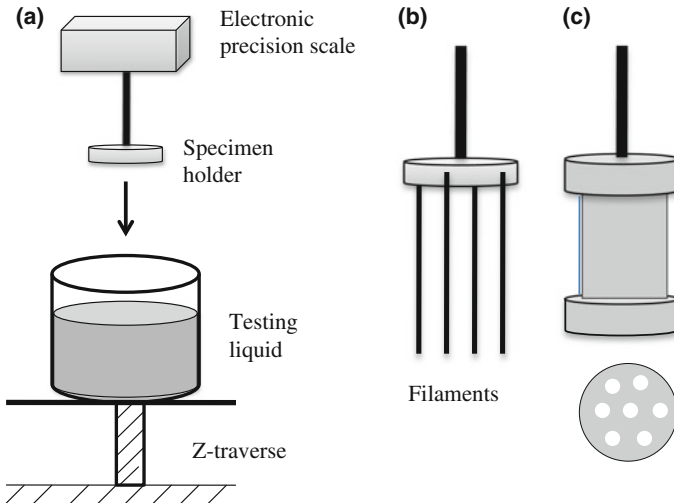


Fig. 16 **a** Wettability measurements setup using a tensiometer. **b** Sample holder for surface tension measurements on single filaments. **c** Sample holder for fiber rovings: the liquid can flow into the sample holder through the holes in the bottom

The method presented above also allows direct measurements of the wetting behavior between fibres and impregnation materials (e.g. sizings or matrix materials), replacing the testing liquid by the impregnation material.

5.4 Adhesion to Matrix

When it comes to reinforcement applications of the fibers, the fiber matrix adhesion is the composite's property which decides on its performance. In this section, at first the principle of load transfer from a matrix to a single fiber is explained. After that, different methods for measuring the fiber matrix adhesion are presented.

5.4.1 Load Transfer and Critical Fiber Length

A parameter which allows the quantification of the fibre-matrix-adhesion (FMA) is the interlaminar shear strength τ_{FMA} . The interlaminar shear strength (ILSS) is the maximum force per surface area with the unit $[N/mm^2]$. In other words: when the shear stress between fiber and matrix exceeds the interlaminar shear strength, a delamination of the interlayers will take place [46] (see Fig. 17).

When stress is transferred from the matrix into a fiber, to general failure behaviours can occur:

- (1) Pull-Out of the fiber: the interlaminar shear strength is lower than the tensile strength of the fiber. Therefore, a delamination takes place and, due to debonding, no more stress can be transferred into the fiber. The reinforcement of the fibers is only possible up to a force which does not exceed the interlaminar shear strength.
- (2) Fiber breakage: the matrix can conduct more stress into the fiber than the fiber's tensile strength can support. Hence, the fiber breaks into smaller pieces

Fig. 17 Definition of the interlaminar shear strength (ILSS), illustrated for the case of a pull-out test

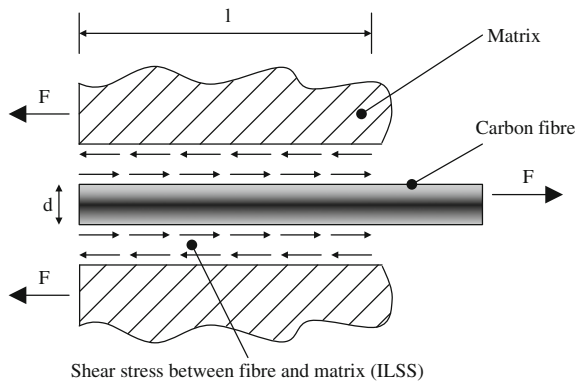
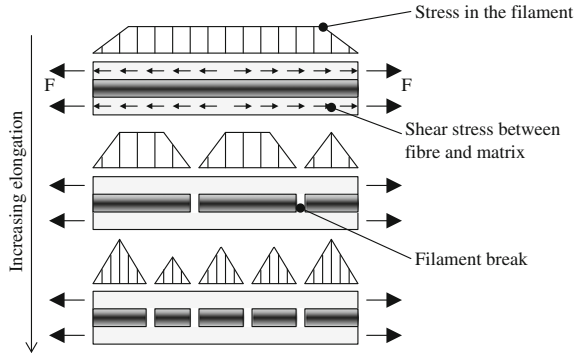


Fig. 18 Stress and fracture development for the case of a composite's elongation: load is transferred to the fibers until the critical fiber length is reached by filament breakage



and the fiber's reinforcement is ideally used until the critical fiber length (length of the fiber where no more stress can be transferred from the matrix to the fibers) is reached (see Fig. 18).

No more reinforcement is possible when the shearing force

$$F_{shear} = A_{surface} \cdot \tau_{FMA} = \pi \cdot d \cdot \frac{l_c}{2} \cdot \tau_{FMA} \quad (11)$$

is at the same level as the maximum tensile force the fiber can withstand:

$$F_{max, fiber} = A_{fiber} \cdot \sigma = \pi \cdot \left(\frac{d}{2}\right)^2 \cdot \sigma \quad (12)$$

Equalizing the two equations leads to the critical fiber length l_c :

$$l_c = \frac{\sigma}{2\tau_{FMA}} \cdot d \quad (13)$$

5.4.2 Overview on Measurement Methods for Fiber Matrix Adhesion

In order to determine the interlaminar shear strength between fibre and matrix, different testing methods (both direct and indirect) have been developed. In real samples, the interlaminar shear stress always occurs in a combination with other stresses, such as tensile stress, pressure and torsion. Nevertheless, the capacitance of fibre reinforced plastics depends significantly on the interlaminar shear strength.

In general, testing methods for the determination of the ILSS can be divided into two groups: direct and indirect testing methods (see Fig. 19).

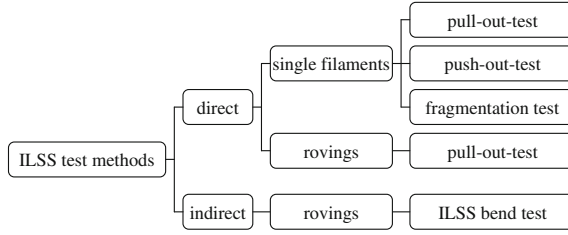


Fig. 19 Overview on testing methods for determining the interlaminar shear strength

5.4.3 Roving Tests

The industrially most commonly used test for characterizing the fiber matrix adhesion is the tree-point-bending test according to ISO 14130:1997 “Fibre-reinforced plastic composites—Determination of apparent interlaminar shear strength by short-beam method” [47]. The sample and measurement setup is shown in Fig. 20.

In the setup shown for the three-point-bending test, a specimen with length l , height h and depth d is set on two supporting axis. In the middle between the two axis, a force F is applied to the specimen until the sample breaks at the force $F_{\max}$. The interlaminar shear strength can be calculated with the data obtained from the test by using the equation:

$$\tau \cong \frac{3 \cdot F_{\max}}{4 \cdot d \cdot h} \quad (14)$$

The advantages of this method are that it can be carried out quickly with standard testing machines. The constraints of this method are that the results obtained are no absolute values. Pure shear stress is only applied in the middle of the sample during testing and the results dramatically change according to the textile structure, the fiber-volume content and the impregnation method used. Hence, the value for τ obtained in this test is called the “apparent” interlaminar shear strength and can be only compared with results obtained with the same setup and the same sample preparation (textile structure, impregnation method).

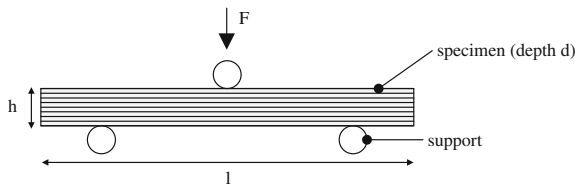


Fig. 20 Measurement setup for three-point-bending test according to ISO 14130:1997

Other common and direct testing method for the interlaminar shear strength is the pull-out test on a roving scale [48]. Therefore, the roving is embedded into resin block on both sides. Between the blocks, the roving is not embedded. One of the resin blocks is shorter than the other one so that the contact area is smaller. When a constantly increasing force pulls the ends away from each other, the roving end in the smaller resin block will delaminate at a certain force level. As the embedded length l_{emb} of the fibers in the shorter resin block, the number of filaments n in the roving and the filament diameter D is known, the interlaminar shear strength can be calculated with the equation:

$$\tau = \frac{F_{max}}{n \cdot \pi \cdot D \cdot l_{emb}} \quad (15)$$

The constraint of this method is that the impregnation has to be perfect i.e. every filament in the roving is surrounded by matrix material on the whole embedded length. Additionally, the roving has to be embedded perfectly straight in the matrix block; otherwise the real embedded length will be different from l_{emb} which was used for the calculation.

5.4.4 Single Filament Tests

Other tests for determining fiber matrix adhesion are employing single filament samples: the single filament pull-out test, the fragmentation test and the single-filament-push-out test.

The single filament pull-out-test and its sample preparation is carried out in the same way as the pull-out-test on roving scale [49].

Within the single filament fragmentation test, a filament is completely embedded into a transparent matrix material. With a special apparatus, the whole specimen is stretched with a constant rate of elongation [50]. Therefore, in the interface between matrix material and fibre surface, a constant shear stress is occurring. If the adhesion between fiber and matrix is weak, it will directly come to delamination. If the fiber matrix adhesion is strong, the shear stress in the boundary layer can induce tensile load into the fiber. If the tensile stress exceeds the tenacity σ of the fibre, the filament breaks into two parts. With further elongation, the effect is reproduced within all fracture pieces remaining until the length of the fiber pieces are shorter than the critical fiber length l_c and no more load can be transferred [51].

The testing procedure is carried out under a light microscope with polarized light while photos of the specimen are taken. The load transfer which has been taking place between fiber and matrix material can be observed by micro cracks (see Fig. 21). The micro cracks appear in white colour. Also the fractures of the fiber can be observed. The microscope images of the elongated samples allow a quantitative comparison of specimen with different grades of fiber matrix adhesion by taking into account the form of the micro crack area.

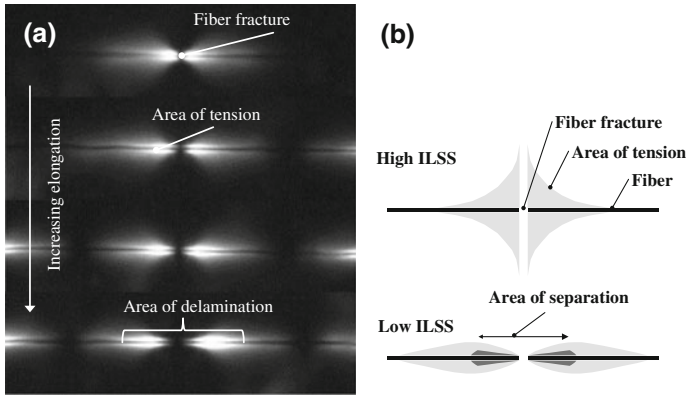


Fig. 21 **a** Microscope images of delamination. **b** Schematic illustration of tension distribution in fiber reinforced composites with different values of ILSS

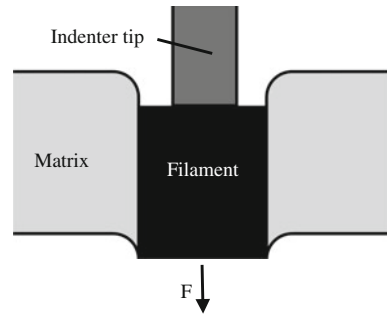
By counting the number of fractures at different elongation stages, the critical fiber length can be determined and the interlaminar shear strength can be calculated according to Eq. (13).

Both the single filament pull-out test as well as the single filament fragmentation test require an effort in sample preparation as in both cases, single filaments have to be prepared out of a roving. As the effects due to the presence of other filaments is neglected in these testing methods, the values obtained for the interlaminar shear strength can be taken as a characterization of the fibers' surfaces, but not directly intend the performance of the material as reinforcement material for composite applications.

The single filament push-out test allows the characterization of a single filament's adhesion to a matrix system in the presence of other filaments. Hence, the conditions of a real composite material are maintained. Moreover, even a detailed investigation of the composite's mechanical properties and crack propagation is possible [52]. The sample preparation (thin sections of approx. 100 μm) is very complex, but on one sample, a nearly unlimited number of single filament push-outs can be carried out.

The principle of the single filament push-out can be seen in Fig. 22. A thin section of a fiber reinforced composite is layed above a substrate with a cavity. The set-up is put into the testing chamber of a nanomechanical testing device. The nanoindenter approaches the sample's surface and the indenter tip pushes out the single filaments into the cavity. The force applied for the push-out is measured as a function of displacement of the indenter tip [53].

Fig. 22 Measurement principle for a single filament push-out-test



6 Physical Properties

In this section, further physical properties of fibres are described together with suitable analytical methods for measuring them.

6.1 Density

The density of a reinforcement fibre is an important measure, because it is the main factor determining the fibre's potential for lightweight construction beside its mechanical properties [9]. Furthermore, the measurement of density is an important tool for quality control in fibre production, because it can be used to determine whether defects are present (e.g. pores leading to a lower density) or production processes are completed (e.g. density increase in carbon fibre conversion) [3].

For the density measurement of fibres, several methods can be applied as indicated in Fig. 23. The following methods are suitable:

- (a) *Density gradient column*: The method is suitable for a direct measurement of the density. Two miscible liquids with different densities are mixed in a column. Due to gravitational effects, a density gradient is found in the column. By putting floats with defined density into the column, a density index can be marked on the column. If fibers with a certain density are put into the column, they sink to the place with corresponding density and start to float, so that their density can be obtained from the scale [54]. The method is very precise if high columns and the right liquids are used for a specific fiber material. However, it can mainly be used for polymeric fibers or other fibers with low density because of the availability of suitable liquids [55].
- (b) *Archimedes' principle*: The density is determined by measuring mass and volume of the fibres. Fibres with a defined mass m are put into a sample holder mounted on a precise scale, which is lowered to a liquid with a defined density ρ_1 (lower than the fiber's density). By measuring the mass in the liquid m'

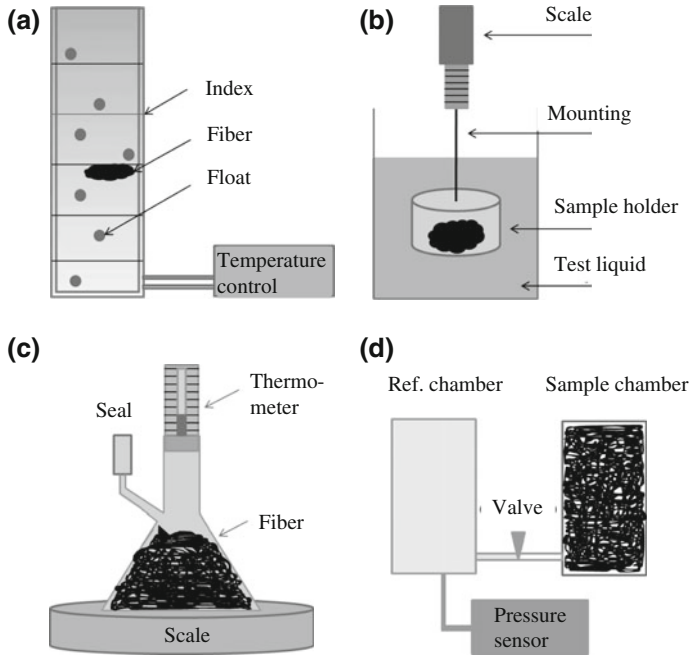


Fig. 23 Methods for measuring the density of fibers. **a** Density gradient column method [54]. **b** Archimedes' principle. **c** Pycnometry (with liquids). **d** Gas pycnometry

(mass change due to the buoyancy force), the fiber's density can be calculated [54, 55]:

$$\rho = \frac{m}{m - m'} \cdot \rho_l \quad (16)$$

- (c) Pycnometry: Fibers with a defined mass m are put into a pycnometer with a precisely defined volume V , which is then filled with a liquid with known density ρ_l . By using a thermometer, the liquid's density can precisely be determined from corresponding tables as a function of temperature. By measuring the total mass of fibers and liquid M in the totally filled pycnometer, the density of the fibers can then be calculated from the mass difference to the pycnometer filled only with liquid [54]:

$$\rho = \frac{m}{V \cdot \rho_l - (M - m)} \rho_l \quad (17)$$

- (d) Gas pycnometry: Gas pycnometry is a very precise method for measuring the volume V of fibres. Together with a precise scale, the density can be obtained. The fibers are filled into a sample chamber with volume V' , which is then evacuated. After evacuation of the chamber, a gaseous medium (usually

helium) is filled into the sample chamber from a reference chamber of the size V'' by opening a valve. By measuring the pressure in the chambers (p before expansion, p' after expansion) and taking into account the ideal gas equation for the test gas, the remaining volume of the test chamber can be obtained and then be used to calculate the fibers' volume [56, 57]:

$$V = V' - \frac{V''}{\frac{p}{p'} - 1} \quad (18)$$

6.2 Thermal Properties

Important thermal properties of reinforcement fibers are described in this section. These are temperatures for phase transitions, heat capacity and thermal conductivity.

6.2.1 Phase Transitions

Phase transitions like glass transition or melting of materials mainly determine the temperature range for their application (e.g. decreasing Young's modulus and tenacity above the glass transition temperature) as well as their processing range (e.g. above their melting temperature for thermoplastic materials) [58].

Phase transitions in fibers are measured by Differential Scanning Calorimetry (DSC). The fiber sample is put into a crucible with good heat contact. The sample and an empty crucible acting as a reference are put onto a sensor with thermocouples (see Fig. 24a). The surrounding chamber is then heated or cooled with a specific temperature profile. By measuring the temperature difference between sample and reference, latent heat in the fiber can be identified (see Fig. 24b). Since the temperature difference is proportional to the heat flow, it can be determined quantitatively by calibrating the method with substances with known melting enthalpy [58].

According to [59], a standard method is available for the determination of transition temperatures. For more specific analysis, special methods with adjusted temperature profiles have to be developed to measure the desired properties. A direct measurement of heat flow is also possible by using so-called power compensating DSC, where two different furnaces (sample and reference) are heated or cooled and differences in power consumption are measured.

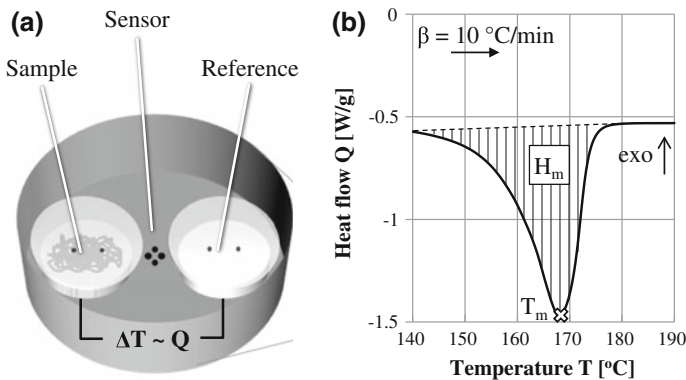


Fig. 24 Differential scanning calorimetry. **a** Sensor for determination of heat flow in sample and reference crucible. **b** Heat flow during the melting of a polymeric fiber

6.2.2 Heat Capacity

The heat capacity c_p describes the amount of energy needed to heat the material by one °C. It can be also determined by DSC, whereas two methods can be applied.

1. The reference crucible is filled with a substance with the same mass and known heat capacity (usually sapphire). By measuring differences in heat flow at temperature ranges with no phase transitions, the heat capacity can be calculated [60].
2. By applying temperature modulation to the standard temperature profile and measuring the time-dependent response of the sample, the reversing part of the heat flow can be identified, which is correlated to the heat capacity [58, 61].

6.2.3 Thermal Conductivity

The thermal conductivity κ of a fiber material describes its property to transfer heat. Due to anisotropic structures, the heat capacity can differ in the directions parallel or perpendicular to the fiber axis (e.g. in carbon fibers, where oriented graphite structures along the fiber increase the thermal conductivity in that direction). Furthermore, the thermal conductivity of textiles or composites is usually better in the direction of oriented fibers, since heat can be conducted along the fibers without heat transfer to other filaments or filament bundles. By adjusting fiber materials and their orientation within the composite, materials with good heat conductivity or thermal insulation in specific directions can be designed.

Single fibres can be tested on their thermal conductivity with special analytical methods. An individual fibre is mounted on a hot wire and temperature changes at the end of the fibre are measured as a function of time [62].

6.3 Thermomechanical Properties

Thermomechanical properties of fibers are important to analyze differences in fiber properties in their application's temperature range. Beside conducting mechanical tests (as described above) at defined temperatures, especially the measurement of thermal expansion (or shrinkage) of the fibers is important. Shrinkage of a fiber can have an impact during the processing of fibers (e.g. in carbon fiber conversion, where draw ratios have to be adjusted) or composites (e.g. by decreasing the fibers' orientation in a textile, which influences the mechanical properties of the composite). Furthermore, internal stress or delamination in the composite part can take place if the thermal expansion of fibers and matrix do not match [1, 9, 10].

The shrinkage behavior of fibers can be measured by standard shrinkage tests or more precisely by thermo-mechanical analysis (TMA). A possible setup is displayed in Fig. 25. Single filaments are fixed at a constant length or force between two clamps and the resulting shrinkage forces or length changes are measured as a function of temperature, while the sample is heated or cooled with a specific temperature profile [63].

6.4 Electrical Properties

The electrical properties of a material can be described with the help of its electrical conductivity σ and dielectrical constant ϵ . Like other physical properties (e.g. thermal conductivity), anisotropic electrical properties can be found in fiber materials due to oriented structures. Depending on the orientation and distribution of fibers, anisotropic electrical properties can also be found in the composite material. The following applications are of great importance:

1. Conductive fibers (especially short or long fibers) are placed in a non-conducting matrix material and can form conductive paths within the material, if a so-called percolating network is formed from the single filaments.

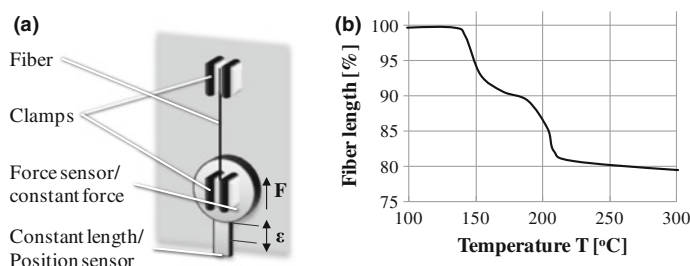


Fig. 25 Thermo-mechanical analysis. **a** Experimental setup. **b** Specific length as a function of temperature for a PAN fiber

- If the fiber volume content passes the percolation threshold, a spontaneous increase of conductivity can be found at this point [64].
2. Anisotropic textile structures (e.g. with uni- or bidirectional fiber placement) made from conductive fibers increase the composite's conductivity in fiber direction. Such structures can be placed in non-conductive composites to remove electrical charges from their surface [65].

6.4.1 Electrical Conductivity

Usually, materials are classified regarding their specific conductivity or their specific resistivity. Resistivity can be measured in the volume (ρ in $\Omega\cdot\text{m}$) or on their surface (R_o in Ω). As displayed in Table 2, the following classes of materials are defined according to [66, 67].

Surface and volume conductivity can be easily measured in composites according to the standards. Anisotropic properties have to be taken into account by measuring in different directions of the material.

For fiber materials, the test procedure has to be adapted. The volume conductivity can only be measured easily in the direction of fibers. Fibers are clamped between two connectors, so that the resistivity of the connections and contacts have to be taken into account. The contact resistivity should be decreased by applying highly conductive material (e.g. silver-based coatings) on the fiber. This procedure is especially important to ensure the contacting of all filaments if fiber bundles are measured. The resistivity values have to be measured at different fiber lengths to allow the calculation of material's conductivity by linear regression [68]. A possible setup for conductivity measurements and the equivalent circuit diagram (taking into account the contact resistivity) are displayed in Fig. 26.

Conductivity tests can be either performed with direct current (DC) or alternating current (AC) at different frequencies. The variation of current frequency allows the investigation if complete conductive paths or single conductive domains only contributing to AC conductivity are present in the fibre [69].

6.4.2 Dielectrical Constant

The dielectrical constant ϵ of a material describes its general behavior in an electrical field. It is usually a complex number ($\epsilon = \epsilon_1 - i\cdot\epsilon_2$) taking into account the

Table 2 Classification of electrical conductivity (according to [66])

Material class	Volume resistivity	Surface resistivity
Conductive	$\rho < 10^4 \Omega\cdot\text{m}$	$R_o < 10^4 \Omega$
Discharging	$10^4 \Omega\cdot\text{m} < \rho < 10^{11} \Omega\cdot\text{m}$	$10^4 \Omega < R_o < 10^{11} \Omega$
Insulating	$10^{11} \Omega\cdot\text{m} < \rho$	$10^{11} \Omega < R_o$

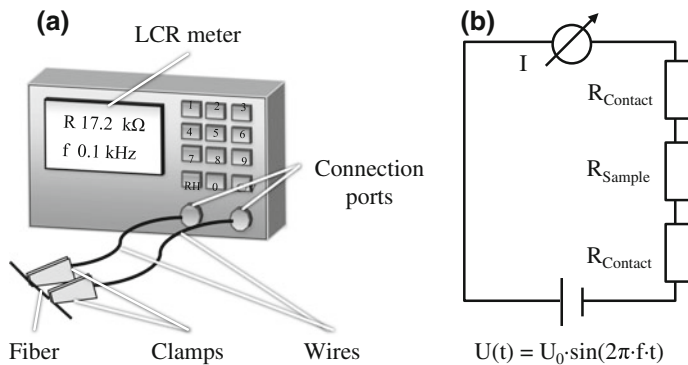


Fig. 26 **a** Experimental setup for measurement of AC resistivity of fiber samples. **b** Equivalent circuit diagram for resistivity measurement of fibers

polarization of the material in an electrical field (real part ε_1) and the dielectrical losses (imaginary part ε_2). Dielectrical losses are an important measure for special applications, e.g. for microwave absorption during composite manufacturing or shielding of electromagnetic fields [70].

Dielectrical properties of a fiber material can be measured by dielectrical spectroscopy. Fibres with a defined geometry are placed between two condensator plates, so that the complex capacity of the condensator with the mounted fibres can be measured as a function of frequency. Taking into account the geometry of the fibres acting as a dielectric between the plates, the dielectrical constant can be calculated.

7 Durability

The durability of fibres has to be taken into account both for the manufacturing of composites as well as during their period of application (even if the fibres are covered with matrix material in the composite, smaller cracks or pores allow the interaction of surrounding media with the fibre). The most suitable test method for all types of durability is to expose to the fibres (or composites) to the specific environmental conditions and test the relevant fibre properties after exposition. For certain applications such as automotive, standardized test methods are available describing the specific environmental conditions. The following types of durability should be taken into account:

1. Thermal stability and degradation: The influence of temperature is present during the curing of the matrix material (especially in thermoplastic matrices or high-temperature matrices) and also in specific applications. Specific temperature ranges to be tested are defined for certain fields of application such as

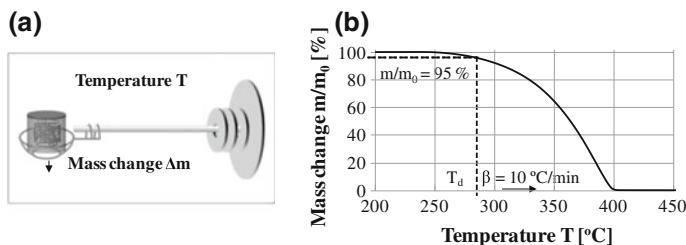


Fig. 27 Thermogravimetric analysis for determination of degradation behavior. **a** Experimental setup. **b** Specific mass as a function of temperature for a polymeric fiber

aerospace or automotive. Especially, the influence on tensile properties should be taken into account. Materials can shrink, melt or degrade at high temperatures or become brittle at low temperatures. Periodic changes in temperature, even if temperature ranges do not exceed certain limits, can lead to structural changes in the fibres which influence the tensile properties.

Temperature ranges to be examined can be identified using DSC (see above, thermal properties) for the identification of phase changes or thermogravimetric analysis (TGA, see Fig. 27a). TGA allows the determination of thermal degradation temperature T_d , which is defined at a relative mass loss of 5 % of the material (see Fig. 27b) [71].

2. Chemical stability: During the curing of matrix systems (e.g. in special systems like concrete), pH values can drastically change from a neutral value. Furthermore, fibre materials may get in contact with alkaline solutions or acids in their fields of application. The stability can be tested by exposing the material to the specific pH value for a period of time and, if necessary, at elevated temperatures. For assessing long-term effects, which may occur after years of application, more harsh conditions can be chosen to study possible effects qualitatively. After exposition, the specific fibre properties can be tested. Degradation due to exposure to chemicals can be tested by determining the fibres weight before and after testing.
3. Mechanical stability: Even if fibres or composites are loaded far below their maximum strength in the linear elastic region, fatigue can occur in the material in the form of plastic deformation, creep or cracks. The behavior can be examined by fatigue tests, where a certain load is applied periodically. Effects on the fibre material and composites can then be studied by the test methods described above [72].

References

1. Wulfhorst B, Gries T, Veit D (2006) Textile technology. Hanser, Munich
2. Elices M, Llorca J (2002) Fiber fracture. Elsevier, Kidlington

3. Morgan P (2005) Carbon fibers and their composites. CRC Press, Boca Raton
4. Pakravan HR, Jamshidi M, Latif M, Pacheco-Torgal F (2012) Influence of acrylic fibers geometry on the mechanical performance of fiber-cement composites. *J App Pol Sci* 125 (4):3050–3057
5. Liu X, Wang R, Wu Z, Liu W (2012) The effect of triangle-shape carbon fiber on the flexural properties of the carbon fiber reinforced plastics. *Mater Let* 73:21–23
6. Liu Y, Chae HG, Choi YH (2015) Preparation of low density hollow carbon fibers by bi-component gel-spinning method. *J Mater Sci* 50:3614–3621
7. Houis S, Schreiber F, Gries T (2008) Fiber-Table according to P.-A. Koch. In: Bicomponent fibres, Shaker, Aachen
8. ASTM D5103—07 (2012) Standard test method for length and length distribution of manufactured staple fibers (Single-Fiber Test)
9. Schürmann H (2005) Konstruieren mit Faser-Kunststoff-Verbunden. Springer, Berlin
10. Ehrenstein GW (2006) Faserverbund-Kunststoffe. Hanser, Munich
11. DIN 53811: 1970–07. Prüfung von Textilien—Faserdurchmesser-Messung in Mikroprojektion der Längsansicht
12. DIN EN ISO 1973: 1995–12. Textilien—Fasern—Bestimmung der Feinheit—Gravimetrisches Verfahren und Schwingungsverfahren
13. Eichhorn S, Hearle JWS, Jaffe M, Kikutani T (2009) Handbook of textile fibre structure: fundamentals and manufactured polymer fibres, vol 1. CRC Press, Boca Raton
14. Salem DR (2000) Structure formation in polymeric fibers. Hanser, Munich
15. Steinmann W, Vad T, Seide G, Gries T, Roth G (2012) Simultaneous analysis of structure and orientation in polymeric fibers by wide-angle X-ray diffraction. Paper presented at the MSE2012 materials science and engineering, Darmstadt, 25–29 Sept 2012
16. Steinmann W, Saelhoff AK, Seide G, Gries T (2014) Analysis of structure formation in carbon fibers: a new method for process development. Paper presented at the ISF 2014 international symposium on fiber science and technology, Tokyo, 28 Sept–1 Oct 2014
17. Steinmann W, Seide G, Gries T (2014) How carbon fibers can get stronger: structure based process development. Paper presented at AUTEX 2014: 14th World textile conference, Bursa, 26–28 May 2014
18. Prevorsek D, Oswald H (1990) Melt-spinning of PET and nylon fibers. In: Schultz JM, Fakirov S (eds) Solid state behavior of linear polyesters and polyamides. Prentice Hall, Englewood Cliffs
19. Bennett SC, Johnson DJ (1978) Strength structure relationships in PAN-based carbon fibres. Paper presented at 5th London international carbon and graphite conference, 1978
20. Ziabicki A (1976) Fundamentals of fiber formation. Wiley, London
21. Borchardt-Ott W (2009) Kristallographie: Eine Einführung für Naturwissenschaftler. Springer, Berlin
22. Schafer et al (1990) High-resolution electron microscopy observations of carbon fibre structures. *Acta Polym* 41:515–518
23. ASTM D3822/D3822M—14: Standard test method for tensile properties of single textile fibers
24. Textechno GmbH (2015) FAVIMAT+ ROBOT2 and AIROBOT2. <http://www.textechno.com/index.php/en/fibre-testing-products-65/favimat-robot-products-145>. Accessed 16 Jun 2015
25. ASTM D2343—09 Standard test method for tensile properties of glass fiber strands, yarns, and rovings used in reinforced plastics
26. ASTM D4018—11 Standard test methods for properties of continuous filament carbon and graphite fiber tows
27. Anderson J (2015) Tensile strength of single fibers: test methods and data analysis. http://cost-fp0802.tuwien.ac.at/fileadmin/mediapool-cost/Diverse/Stockholm_Workshop/Andersons.pdf. Accessed 16 Jun 2015

28. Asad RAM, Yu W, Zheng Y, He Y (2015) Characterization of prickly tactile discomfort properties of different textile single fibers using an axial fiber-compression-bending analyzer. *Text Res J* 85(5):512–523
29. Naito K, Tanaka Y, Yang JM, Kagawa Y (2009) Tensile and flexural properties of single carbon fibres. Paper presented at ICCM 17 international committee on composite materials, Edinburgh, 27–31 July 2009
30. Saelhoff AK, Jäger M, Steinmann W, Gries T (2014) Surface treatment of carbon fibers—increasing the interlaminar shear strength in CFRP. Paper presented at the ADITC2014 Aachen-Dresdner international textile conference, Dresden, 27–28 Nov 2015
31. Steinmann W, Wulforth J, Walter S, Seide G, Gries T (2012) Nanoparticles in polymeric fibers: novel possibilities for the modification of surface, mechanical and electrical properties. Paper presented at the nanoscience conference, Yaiza, 14–17 Feb 2012
32. Rosiepen C, Beck T, Hehl A, Gries T (2012) Tribology of textiles: challenging carbon fibre processing. Paper presented at the MSE2012 materials science and engineering, Darmstadt, 25–29 Sept 2012
33. ISO 9277:2010: Determination of the specific surface area of solids by gas adsorption—BET method
34. Braunaue S, Emmet PH, Teller E (1938) Adsorption of gases in multilayered layers. *Am Chem Soc* 60(2):309–319
35. Binnig G, Quate CF, Gerber C (1986) Atomic force microscope. *Phys Rev Lett* 56(9):930–933
36. Cardona M, Ley L (eds) (1979) Photoemission in solids I, II. *Topics Appl Phys* 1:26–27 (Springer, Berlin)
37. Hüfner S (1996) Photoelectron spectroscopy, principles and applications. *Solid-State Sciences*, vol 82. Springer, Berlin
38. Benninghoven A, Rüdenauer FG, Werner HW (1987) Secondary ion mass spectrometry: basic concepts, instrumental aspects, applications, and trends. Wiley, New York
39. Liebl H (1967) Ion microprobe mass analyzer. *J Appl Phys* 38:5277–5280
40. Adamson AW, Gast AP (1997) Physical chemistry of surfaces. Wiley, Hoboken
41. Ström G, Fredriksson M, Stenius P (1987) Contact angles, work of adhesion, and interfacial tensions at a dissolving Hydrocarbon surface. *J Colloid Interface Sci* 119:352–361
42. Fowkes FM (1964) Predicting attractive forces at interfaces. *Ind Eng Chem Res* 56:40–53
43. Hoecker F, Karger-Kocsis J (1996) Surface energetics of carbon fibers and its effects on the mechanical performance of CF/CP composites. *J Appl Polym Sci* 59:139–153
44. Washburn EW (1921) The dynamics of capillary flow. *Phys Rev* 17:273
45. Bruil HG, Aartsen JJ (1974) The determination of contact angles of aqueous surfactant solutions on powders. *Colloid Polym Sci* 252:32–38
46. Drazal L, Madhukar M (1993) Fibre-matrix adhesion and its relationship to composite mechanical properties. *J Mater Sci* 28:569–610
47. ISO 14130:1997—Fibre-reinforced plastic composites—determination of apparent interlaminar shear strength by short-beam method
48. Michaeli W, Fölster T, Klink R, Kocker K (1993) Faserbündel-Pull-Out-Versuch. *Kunststoffe* 83:65–69
49. Bannister DJ, Andrews MC, Cervenka AJ, Young RJ (1995) Analysis of the single-fibre pull-out test by means of Raman spectroscopy: part II. *Compos Sci Technol* 53:411–421
50. Feih S, Wonsyld K, Minzari D, Westermann P, Lilholt H (2004) Testing procedure for the single fiber fragmentation test. Risø National Laboratory, Roskilde
51. Netravali AN, Henstenburg RB, Phoenix SL, Schwartz P (1989) Interfacial shear strength studies using the single-filament-composite test. *Polym Compos* 10(4):226–241
52. Jäger J, Sause MGR, Burkert F, Moosburger-Will J, Greisel M, Horn S (2015) Influence of plastic deformation on single-fiber push-out tests of carbon fiber reinforced epoxy resin. *Compos A* 71:157–167
53. Greisel M, Jäger J, Moosburger-Will J, Sause MGR, Mueller WM, Horn S (2014) Influence of residual thermal stress in carbon fiber-reinforced thermoplastic composites on interfacial fracture toughness evaluated by cyclic single-fiber push-out tests. *Compos A* 66:117–127

54. ISO 10119-2 Carbon fibre—determination of density
55. Truong M, Zhong W (2009) A comparative study on natural fibre density measurement. *J Text Inst* 100(6):525–529
56. GmbH Micromeritics (2010) Highly adaptable density determinations—the AccyPyc II 1340 gas displacement pycnometric system. Micromeritics GmbH, Mönchengladbach
57. DIN 51913 Bestimmung der Dichte mit einem Gaspyknometer (volumetrisch) unter Verwendung von Helium als Messgas
58. Steinmann W, Walter S, Beckers M, Seide G, Gries T (2013) Thermal analysis of phase transitions and crystallization in polymeric fibers. In: Elkordy AA (ed) Applications of calorimetry in a wide context: differential scanning calorimetry, isothermal titration calorimetry and minicalorimetry. InTech Europe, Rijeka, pp 277–306
59. ASTM D3418 Standard test method for transition temperatures and enthalpies of fusion and crystallization of polymers by differential scanning calorimetry
60. Takahashi Y (1985) Latent heat measurement by DSC with sapphire as standard material. *Thermochim Acta* 88(1):199–204
61. Shawe JEK, Hütter T, Heitz C, Alig I, Lellinger D (2006) *Thermochim Acta* 446(1,2): 147–155
62. Wang JL, Gu M, Zhang X, Song Y (2009) Thermal conductivity measurement of an individual fibre using a T type probe method. *J Phys D* 42:105502–105509
63. Heine M (1988) Optimierung der Reaktionsbedingungen von thermoplastischen Polymer-Fasern zur Kohlenstoffaser-Herstellung am Beispiel von Polyacrylnitril. Universität Karlsruhe, Dissertation
64. Steinmann W, Vad T, Weise B, Wulforth J, Seide G, Gries T, Heidelmann M, Weirich T (2013) Extrusion of CNT-modified polymers with low viscosity—influence of crystallization and CNT orientation on the electrical properties. *J Polym Polym Compos* 21(8):473–482
65. Kilbride M, Pethrick RA (2012) Enhancement of the surface electrical conductivity of thermoplastic composite matrices. *J Mater Des Appl* 226(3):252–264
66. Bundesanstalt für Arbeitsschutz und Arbeitsmedizin (BAuA) (2009) Vermeidung von Zündgefahren infolge elektrostatischer Aufladungen. BAuA, Dortmund
67. ANSI/ESD S541-2008 For the protection of electrostatic discharge susceptible items: packaging materials for ESD sensitive items
68. Steinmann W (2014) Elektrisch leitfähige Fasern aus Polymer-Nanoverbundwerkstoffen. RWTH Aachen, Dissertation
69. Glauß B, Steinmann W, Walter S, Beckers M, Seide G, Gries T, Roth G (2013) Spinnability and characteristics of polyvinylidene fluoride (PVDF)-based bicomponent fibers with a carbon nanotube (CNT) modified polypropylene core for piezoelectric applications. *Materials* 6 (7):2642–2661
70. Elimat ZM, Hamideen MS, Schulte KI, Wittich H, de la Vega A, Wichmann M, Buschhorn S (2010) Dielectric properties of epoxy/short carbon fiber composites. *J Mater Sci* 45: 5196–5203
71. ASTM D3850 Test method for rapid thermal degradation of solid electrical insulating materials by thermogravimetric method (TGA)
72. Gude M, Hufenbach W, Koch I, Koschichow R, Schulte K, Knoll J (2013) Fatigue testing of carbon fibre reinforced polymers under VHCF loading. *Procedia Mater Sci* 2:18–24

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