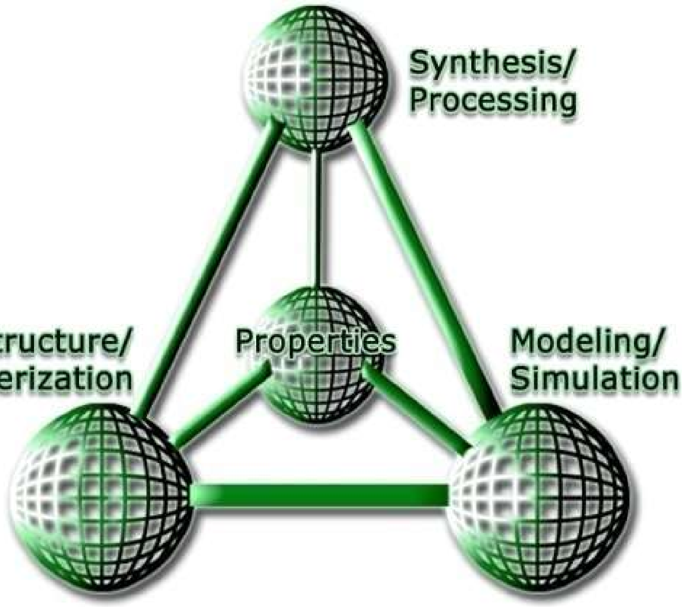
Recent Trends in Fibrous Material Science



Volume - V
September - 2019

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RECENT TRENDS IN FIBROUS MATERIAL SCIENCE

Liberec 2019

Chapter 8

Surface Modification of Textile Fibers by Whiskerization

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1. INTRODUCTION

Textiles fibers have been used for humans' purposes since antiquity. Firstly, fur and animal skin had been used for dressing and protection from the environment, then they started to use vegetables fibers to make rudimentary fabrics. There are evidences of the use of dyed flax fibers into clothes more than 30,000 years ago. For decades and centuries, humans have used natural fibers coming from both plants and animals (such as flax, hemp, cotton, wool...etc.) to produce yarns strands and then weave them into fabrics using manual processes. In the eighteenth century, there was a revolutionary industrial development with the invention of machines and equipment that accelerated the manufacture of fabrics and made them more available and economically feasible. Such industrial revolution changed the concept of textile manufacturing and transformed it into a real industry. In the twentieth century, it could be said that there was a second technological and industrial revolution with the synthesis of man-made fibers such as rayon, nylon, or polyester with good quality and low-cost. This rapidly gave those fibers a significant market share because of their good properties as well as low cost, chemical stability, and outstanding versatility (dyes, colors, fiber diameters, engineered weaving for special clothes).

Currently, there is a new revolution on the textile industry with the surprising apparition of new technologies that could add special functions and properties to the fabrics. For example, there has been significant improvement in methods for textile coloring, digital printing on textiles, smart fabrics, and high-performance functional textiles. In this sense, nanomaterials play a significant role in this technological evolution since they show outstanding surface properties that allow to multiply their effect in comparison with bulky traditional additives and materials.

This chapter reviews the most relevant contributions of growing Nano-wiskers on the textile fiber surface or whiskerization for functionalize textile materials. Firstly, the chapter will go through the different techniques of growing nanowiskers then will review the applications of nanowiskers on different textile fibers. The review work here will focus on the functionalization of specific textile fibers which are mostly used in the high added value applications such as carbon fiber and glass fiber, as well as natural fibers such as jute and flax.

2. NANOWISKERS GROWING ON TEXTILE FIBER SURFACE

The inherited characteristics of textile fibers can be improved by growing nanostructured materials on their surfaces, a process commonly known as “whiskerization”. Various methods such as chemical vapor deposition, hydrothermal process and electrochemical deposition are usually used to carry out the growth of nanostructure on the fibers surface. During the growth process, the nanostructures are directly built on the surface layer of the textile fibers, either through the pre-deposited “seed layer” or hydrophobic interaction. After the growth process, the as grown nanostructures and the textile fiber substrates get integrated and forming free-standing, binder-free multi-scale composites. Depending on the properties of the as-grown nanostructures, the applied functions of textile fiber can be greatly enhanced and extended such as antibacterial activity, flame retardant properties, UV-protection, supehydrophobicity, increasing bonding force between the textile fibers and matrix in composite structure and others.

2.1 Nanowiskers Grown on Carbon Fiber

Due to its high mechanical strength, light weight carbon fiber has become one of the most important materials in fabricating structural components and load-bearing parts, and has found many applications such as automotive, sports, building, aircrafts and aerospace shuttles, military application, medical…etc. recognized by its name, carbon fiber is composed of the elemental carbon (C). as distinct from graphite, which is composed of sheets of carbon atoms (graphene sheets) that parallel stack on each other, carbon fibers are composed of graphene sheets that twisted, folded and crumbled upon each other. Therefore, carbon fibers would have high tensile strength and stiffness as compared to graphite. Carbon fibers are produced from the precursors such as polyacrylonitrile (PAN), rayon and petroleum pitch. The precursors are spun and drawn to form filament tows, which are subsequently subjected to three stages, pre-oxidation, thermal carbonization and graphitization processes. After the thermal carbonization process, the precursor filament tows are converted into carbon filament yarns with high carbon contents. The carbon filament yarns produced from the carbonization process generally exhibit high tensile strength while the ones produced from the graphitization process generally exhibit high elastic modulus. Due to its high performance characteristics such as mechanical strength, high modulus, thermal conductivity and low thermal expansion, carbon fibers are extensively used in the high technology sectors, such as aerospace and nuclear engineering, where high performance under high damping, high temperature and corrosive environment is needed. However, in general engineering sectors and transportation, the application of carbon fiber is constrained by its cost and production rates. Depending on the precursor, the final properties of carbon fiber can be produced based on the specific requirements of application. For example, the carbon fiber produced from PAN has the highest tensile strength, which is suitable for the high technology applications. On the other, the carbon fiber produced from cellulose may have lower tensile strength accompanied with low cost, which is suitable for general engineering applications.

As mentioned before, the characteristics of carbon fibers can be improved by growing nanostructured materials on their surfaces, a process commonly known as "whiskerization". Various methods such as chemical vapor deposition, hydrothermal process and electrochemical deposition are usually used to carry out the growth of nanostructure on the fibers surface.

Carbon nanotubes (CNTs) grown on carbon fibers are speculated to improve the interfacial bonding in FRC due to the high mechanical strength, high surface area and good substrate adhesion [21]. Extensive research efforts have been devoted to grow CNTs on carbon fibers while the diameter, length and crystallinity of the as-grown CNTs can be effectively controlled [22–25]. Techniques of chemical vapor deposition (CVD) are widely applied to grow CNTs on carbon fiber surface (Figure 1). In a typical process, the carbon fibers are firstly cleaned and desized in organic solvents by sonication, and then immersed in the catalyst solution at elevated temperature for absorbing and loading of the metal catalysts. Metals, such as iron (Fe), nickel (Ni), cobalt (Co), are the major catalysts used for the growth of CNTs [27, 28]. After the immersion and the subsequent drying process, the surface of carbon fiber is densely loaded with small metal particles, as shown in Figure 2. Afterwards, the metal-loaded carbon fibers are placed in a quartz tube furnace, which are subsequently heated in the presence of hydrogen (H2) and carbon source mixed stream (e.g., benzene, ethylene, acetylene) to accomplish the CNT growth. The flow rate of total gas streams is typically in the range between 100 mL min1 and 300 mL min^{-1}, and inert gas protection is required during the heating and cooling steps.

The as-grown CNTs exhibit long and curved shapes which wrapped around the longitudinal axis of the carbon fibers randomly, as shown in Figure 3. The morphology of the as-grown CNTs can be controlled by a wide-range of parameters including types of catalyst, catalyst concentration, gas flow rate, growth time and temperature. A brief summary of the relationship between the carbon fiber morphology and these parameters is provided in Table 3. Amongst these parameters, the catalyst concentration is speculated to play a major role since all the morphological related parameters (e.g., diameter, length, density) can be effectively tuned by it (Figure 4). It should be noted that there exists a proper range for tuning the growth parameters of CNTs, and beyond this range CNTs may not be properly grown [30]. Other than CNTs, carbon nanofibers (CNFs) can also be grown on the surface of carbon fiber by using the same chemical vapor deposition (CVD) procedures [31–33]. Different from CNTs, the CNFs is characterized as long nanofibers with solid core, and they would generally have higher aspect ratios than CNTs. It is speculated that by increasing the time of CVD the CNTs can be further grown into CNFs, as shown in Figure 5 [34].

Metal oxide nanostructures can be grown on carbon fibers surface to improve their performances such as interfacial bonding strength in composites structures. Hydrothermal technique is the method which is used to grow metal oxide nanostructures, as illustrated in Figures 3 and 4. Growing metal oxide nanostructures on carbon fibers by the hydrothermal method may have the advantages including:

(i) High degree of morphological control over the as-grown nanostructures can be achieved on the carbon fiber surface. In other words, high structural uniformity and high growth density can be readily achieved for the as-grown metal oxide nanostructures.
(ii) Growth process of metal oxides is simpler and requires less instrumentation, the material and energy consumption are also less comparing with the thermal CVD process.
(iii) by using the same growth protocol, different types of nanostructured metal oxides can be grown on the surface of carbon fibers.

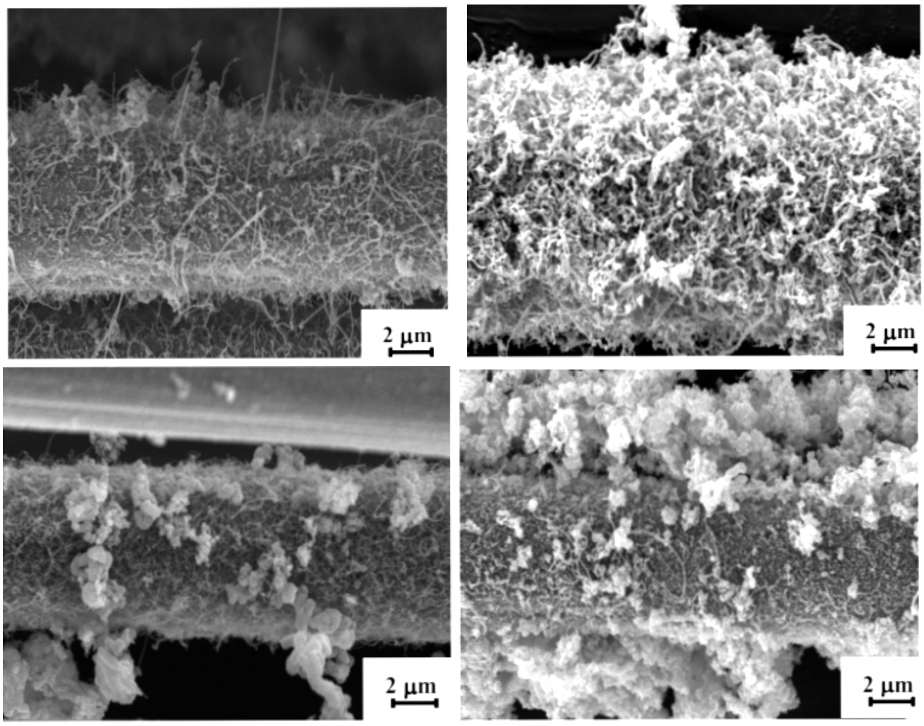


Fig. 1 CNTs grown on carbon fibers under different reaction parameters [4]

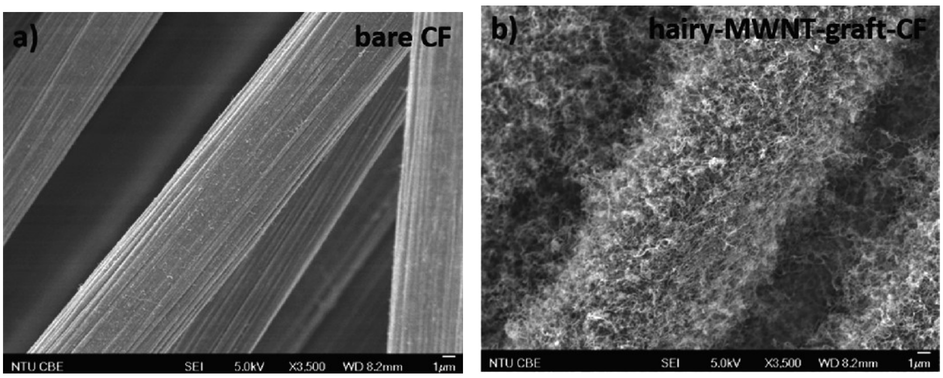


Fig. 2 Carbon fibers SEM images (a) before growing (b) after growing MWNT by CVD (5)

However, CNTs and CNFs grown by thermal CVD may still possess the pros including:

(i) higher theoretically predicted improvement in the interfacial strength for FRC;
(ii) higher surface area of the as grown nanostructures, and (iii) better affinity or adhesion to the carbon fiber substrate.

(a) (b)

Fig. 3 Laboratory autoclave vessel (a) schematic diagram (b) Photograph [6]

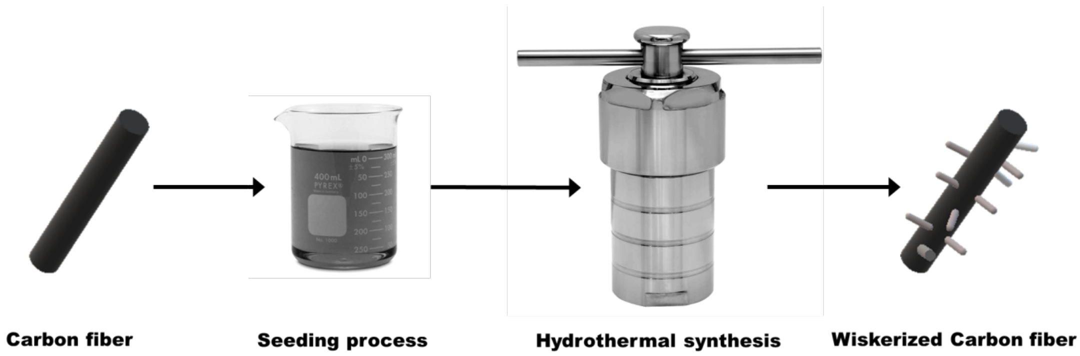


Fig. 4 Schematic diagram of a typical hydrothermal process that used for the synthesis of metal oxide on carbon fiber surface

In hydrothermal metal oxides synthesis process, firstly the metal oxide “seeds” are deposited on the surface of carbon fiber by immersing the carbon fibers in the solution of metal salts. then, the seed-loaded carbon fibers are annealed at elevated temperature in atmospheric pressure, in order to improve the adhesion between the seeds and the fibers. Then the treated carbon fibers are immersed in a “growth solution” which contains the metal salts and organic polyamines (e.g., hexamethylenetetramine, HMTA). The growth process is then propagated by heating the solution in a glass beaker at elevated temperature on a hotplate. Or a stainless-steel autoclave can be used if higher temperature and pressure are needed figure 3. The zinc oxide (ZnO), Zirconium carbide (ZrC)) nanowires, titanium dioxide (TiO2) nanorods and nanosheet and Fe_2O_3 nanorings synthesized by using the hydrothermal method are shown in Figure 5. Similar to the growth of nanocarbons, structural control over the as-grown metal oxide nanostructures can be achieved by tuning the concentration of the “seeding solution,” loading quantity of the metal oxide “seeds,” as well as the time of growth (Figure 5).

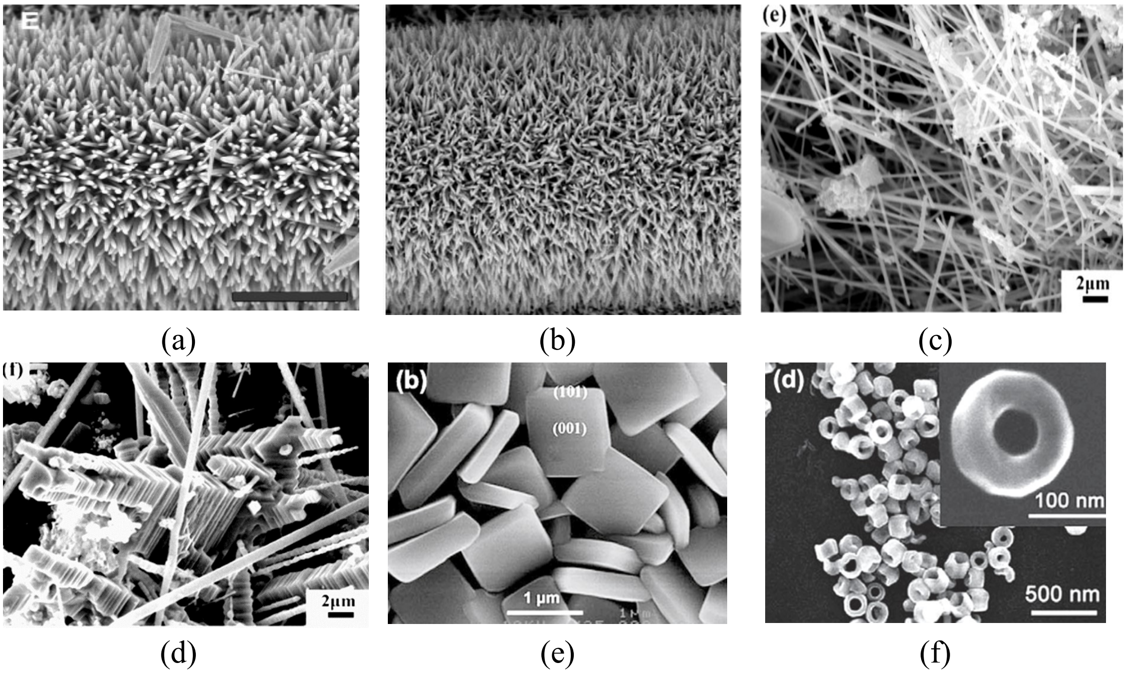


Fig. 5 SEM images of different metal oxides at different synthesis parameters (a and b) ZnO nanowiskers, (c and d) ZrC nanowhiskers, e) Anatase TiO_2 nanosheets, f) Fe_2O_3 nanorings [8, 9]

In the work of Galan, et al, growth parameters were controlled to achieve ZnO nanowires with diameters ranging from 60 nm to 160 nm and lengths from 0.1 um to 1.1 um. Nanowire diameter was controlled by adding PEI to the growth solution while all other parameters fixed (chemical concentration, temperature, ZnO nanoparticle size, etc.). Nanowire length was influenced by all these parameters and length increased as growth time was extended as it can be seen from figure 6.

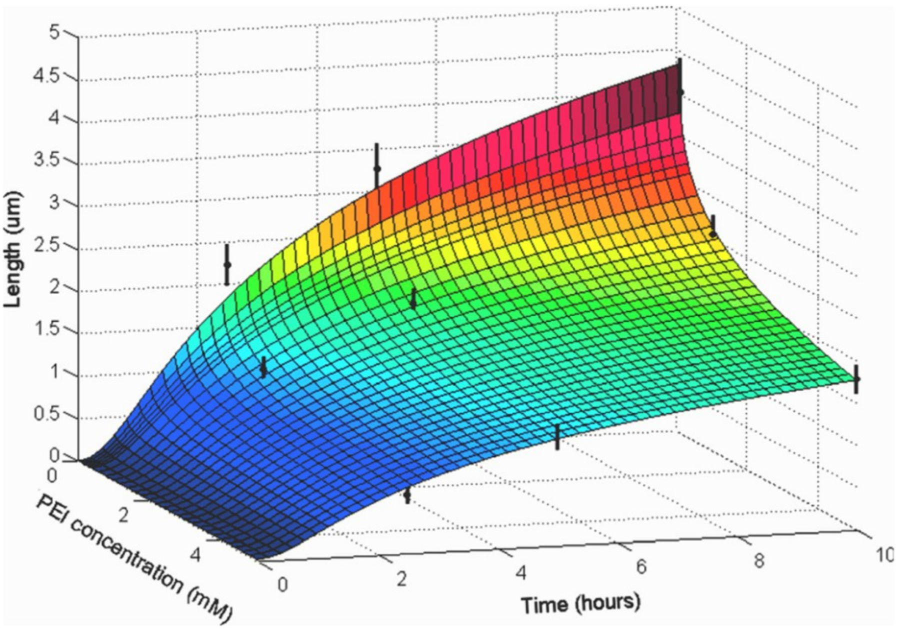


Fig. 6 Length of ZnO nanowires as a function of PEI concentration and growth time [7]

3. EFFECT OF WHISKERIZATION ON THE MECHANICAL PROPERTIES OF CARBON FIBER AND CARBON FIBER REINFORCED COMPOSITES

The carbon fibers wiskerized with carbon or metal oxide nanostructures on the surface are subject to the mechanical testing to reveal their functional performance in enhancing the interfacial bonding strength between the fiber and matrix within the FRC, consequently increasing the mechanical strength of the whole FRC. As it can be noticed from Table 1, It is found that CNTs and CNFs are able to increase the mechanical strength of the carbon fiber reinforced composites (CFRC) to a great extent, while the metal oxide nanowires can significantly improve the interfacial strength between the carbon fiber and polymer matrix.

Table 1. Mechanical properties of carbon fiber with nanostructured materials grown on its surface [3, 10, 11, 12, 13, 14, 15]

Materials	Tensile strength (MPa)	Modulus (GPa)	Interfacial shear strength (MPa)	Maximum increment (%)
CNT-CF	27	1.07		133% in tensile strength
ZnO NW		3.34	33.87	113% in shear strength
CNF	23.9–24.8	0.75–0.79		
CNT				17% in fracture toughness
CuO NW				42.8% in tensile strength
TiO_2 NR	200.5			45% in tensile strength
ZnO NW				209.5% in loss factor
ZnO NR				50% in loss factor
CNT				300% in conductivity
CNT				510% in conductivity
CNT				56% in loss factor
CNT				69% decrease of crack propagation
CNT			18.1	45% in shear strength
CNT				127% in impact energy dissipation
CNT				30% in shear strength
SiO_2 NP			52	44% in shear strength
Graphene				173% in shear strength

NW nanowires, NR nanorods, NP nanoparticles, CNT carbon nanotubes

In the work of Kowbel, et al, [16] Carbon fabric whiskerization via a direct, non-catalytic Sic whisker formation was investigated as a method to increase the shear and transverse properties of carbon-carbon (C-C) composites. The whiskers were produced via a SiO gas reaction with the carbon fiber surface. A variable level of whisker's population density was achieved by varying the whiskerization conditions. The flexure strength of phenolic resin-derived C-C composites made with the whiskerized fabric was found to be decreasing with increasing the extent of the fiber whiskerization. A 300% increase in the interlaminar strength (ILT) combined with a

250% increase in the interlaminar shear strength (ILS) was found in the case of the composites made with a low whiskerized fabric.

Another work has been carried out by Wang et al, [17] in this study, graphene nanowalls (GNWs) with different sizes (i.e., length and height) were grown directly on the surface of individual carbon fibers (CFs) using a radio frequency plasma-enhanced chemical vapor deposition (RF-PECVD) technique. The size was controlled by varying the deposition time. The GNW-modified CFs were embedded into epoxy resin matrix to prepare a series of carbon-fiber-reinforced composites (CFRCs). The results indicated that GNWs were remarkably effective in improving the interfacial shear strength (IFSS) and interlaminar shear strength (ILSS) of the carbon-fiber-reinforced composites. The enhancement effect on the strength strongly depended on the size of GNWs. It increased with the increase in the GNWs' size and reached the maximum upon the incorporation of GNWs that were grown for 45 min. Noticeable increases of 222.8% and 41.1% were observed in IFSS and ILSS, respectively. The enhancement mechanism was revealed by means of scanning electron microscope (SEM) fractography analysis. However, further increase of GNW size led to no more improvement in the shear strength. It could result from the increased defect concentration and wrinkle size in the GNWs, which deteriorated the strength**.**

Vishkaei et al [18] had been investigated the effects of whiskerized carbon fibers (WCF) embedded as filler into polymer matrix were investigated. In this respect, composites consisting of pure polypropylene and also carbon fiber (CF)/polypropylene (PP) was fabricated and compared. Polypropylene matrix was reinforced with 2% concentration of WCF and prepared by a melt-mixing method. The tensile test indicated that the addition of 2% WCF enhanced the tensile strength and Young's modulus by 38.1% and 28.2%, respectively. Besides that, the elongation was decreased for that sample. Dynamic mechanical analysis showed an increase of 39.2% in the stiffness of the WCF/PP composite and an improvement in the storage modulus. The tan d for the sample was also smaller than unfilled PP and CF/PP composites. Furthermore, thermogravimetric analyses in an inert atmosphere showed a shift of temperature to the higher temperature with the addition of fillers.

Galan, et al [7] have developed the parameters for the growth of nanowires with varying lengths and diameters and study the influence of the nanowire's morphology on the interfacial shear strength. ZnO nanowire arrays are grown on carbon fibers, with nanowire diameters ranging from 50 to 200 nm and lengths up to 4 um. The interfacial shear strength with varying nanowire dimensions is shown to increase by up to 228%, ranging from 45.72 to 154.64 MPa. Unlike existing whiskerization approaches, it is shown that the tensile strength of the ZnO nanowire coated fibers remains constant throughout all growth procedures. The development of an inter-phase offering control over the interface strength and toughness will provide a means to produce multifunctional composites with optimized performance for multiple applications.

In the work of Majumdar, et al [19] Zinc oxide (ZnO) nanostructures have been grown on p-aramid (Kevlar) fabric to improve interfacial properties of reinforced composites. Whiskerization technique was adopted to develop ZnO nanostructures, like an array of whiskers, on the surface of Kevlar fabric. Hydrothermal process was used which involves three steps i.e. functionalization, seed layer formation and growth of nanorods. Hexamethylenetetramine and zinc nitrate were used to grow ZnO nanostructures. It was found that pressure-controlled autoclave was the best method for generation of nanostructures. It was also observed that the material (fabric) to solution ratio (M:S) significantly affected the morphology and density of developed nanostructures. Incorporation of ZnO nanostructures on Kevlar fabric improved the tensile and impact properties of Kevlar fabric and its composites. The deposition of nanostructures improved the interfacial properties by causing fibers to interlock with matrix thus allowing the formation of a graded interface between the two phases.

Zheng, et al, [20] grew ZnO NWs onto carbon fabrics through a facile hydrothermal method, and the pull-off force to detach an individual ZnO nanowire from CF was measured using a nano-manipulator inside a scanning electron microscope chamber. Also, a novel dopamine-based functionalization method was developed to improve the interfacial adhesion between ZnO NWs and CFs. It was found that introducing polydopamine (PDA) on CF could increase significantly the adhesion strength between CF and ZnO NW and their interfacial shear strength with epoxy as measured by the single fiber microbond test. The hierarchical ZnO NWs on CF fabrics were then utilized to fabricate the laminates. The highest mode I and mode II interlaminar toughness were obtained in those laminates comprising CF/PDA/ZnO NWs owing to the high chemical bonding between ZnO NWs and PDA modified CF surface and strong mechanical interlocking between ZnO NWs and epoxy.

Li, et al, [21] Silicon nitride (Si_3N_4) nanowires were in-situ fabricated on carbon fiber fabrics up to 5 cm 11 cm in area via catalyst-assisted pyrolysis of polymeric precursors. The obtained products were randomly oriented around the carbon fibers with the diameter of 30–150 nm and the length of several hundred micrometers. The effects of process parameters including the preparation temperature, flow rate of nitrogen, catalyst proportion, and volume fraction of acetone on the synthesis of Si_3N_4 nanowires were discussed. Accordingly, a set of optimized process parameters was determined. The microstructure of the nanowires indicated that their formation obeyed a solid–liquid–gas–solid (SLGS) growth mechanism.

Kim et al [22] Zinc oxide nanorod (ZnO NR)-grown woven carbon fiber/polyamide 6 composites were fabricated using hydrothermal synthesis and thermoplastic resin transfer molding. The in-situ polymerization of ε-caprolactam, which exhibits extremely low viscosity and high reaction speed, enabled excellent penetration of the resin into the densely grown ZnO forest on the carbon fibers. This further increased the mechanical interlocking and chemical interaction between the fiber and resin, leading to enhanced interfacial bonding. By increasing the number of oxygen functional groups and the surface roughness of the fiber surfaces through an atmospheric plasma treatment, the ZnO NRs were observed to grow even with very low growth-solution concentrations (20 mM), fewer seed cycles (4), and a short hydrothermal treatment time (4 h). By using the plasma-treated carbon fibers for ZnO NR growth, the impact resistance and in-plane shear strength were enhanced by up to

72 and 50%, respectively, as compared to carbon fiber composites without ZnO NRs, while the use of ZnO precursors and growth time was minimized.

4. EFFECT OF WHISKERIZATION ON THE PROPERTIES OF NATURAL FIBER AND NATURAL FIBER REINFORCED COMPOSITES

The recent developments of fiber engineering are towards the growth and usage of natural in many fields of engineering due to both environmental and economic benefits. Due to the low cost of natural fibers and their high specific strength, they are exploited as a replacement for the conventional man-made fibers, such as glass and carbon. In addition, natural fibers are renewable, easily recycled, carbon dioxide neutral, and are locally available in large quantities all over the world and especially at many under development countries.

In this part of the chapter, authors review the most recent attempts to modify the natural fiber surface by growing nanostructures (whiskerization) to utilize the natural fibers in different engineering fields.

Arfaoui, et al, [23] developed a hydrophobic treatment for jute fibers based on the grafting and growth of ZnO nanorods on fiber surface. The first step consists in removing impurities from the fiber surface with a scouring treatment. the second step is seeding stage, in this stage the jute fibers are coated with ZnO layer nanoseeds. In the third step, hydrothermal process is carried out to ensure a uniform growth of ZnO nanorods on the surface of fibers. Finally, a hydrophobic treatment is performed on the ZnO nanorod-covered jute fibers using stearic acid (SA), i.e., a typical fatty acid. The results showed significant improvement in the fiber hydrophobicity without any negative effect on thermal stability and limited reduction Jute fiber in strength.

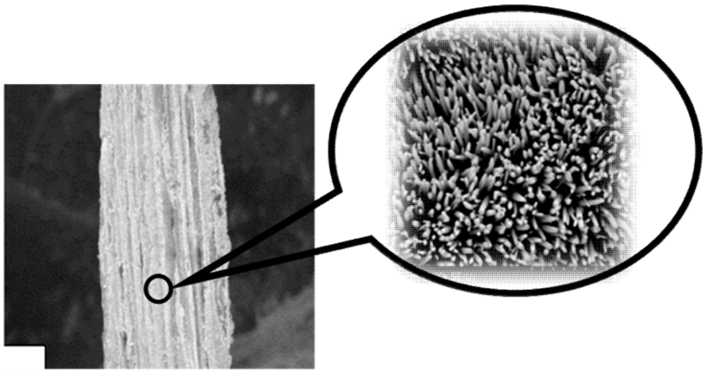


Fig. 7 SEM images of jute fibers and jute fibers coated with ZnO nanorods [23]

Dolez, et al. [24], described hydrophobic treatments developed for natural fibers. They include coating fibers and nonwovens fabrics by metal oxide nanoparticles, namely titanium dioxide (TiO) and zinc oxide (ZnO), then they applied a layer of a fatty acid. The efficiency of treatments was evaluated on recycled jute in terms of water contact angle and water drop shape retention time. In addition, the effect of the hydrophobic treatment process on the mechanical performance and thermal stability was also tested. The results showed the advantage of the metal oxide nanoparticle intermediate layer in providing the natural fibers with stable hydrophobic properties. These treatments provide a nontoxic, low cost solution to make natural fibers hydrophobic, including recycled ones. This opens new opportunities for these fibers as reinforcement for composite parts.

In our work [25], hierarchical ZnO nano and microwhiskers were grown for directly the first time on a bacterial cellulose substrate and on two additional different types of papers by hydrothermal synthesis without any surface modification layer (fig. 8 and 9). Compactness and smoothness of the substrates are two important parameters that allow the growth of oriented structures.

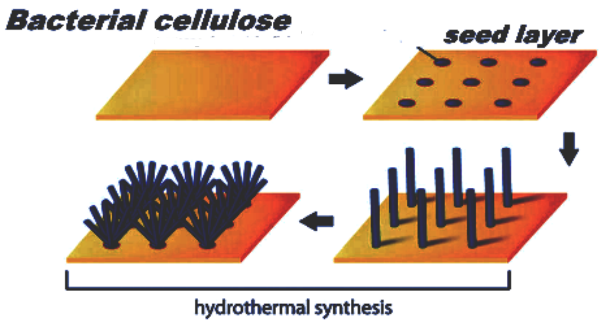


Fig. 8 ZnO microflower and nanorods preparation

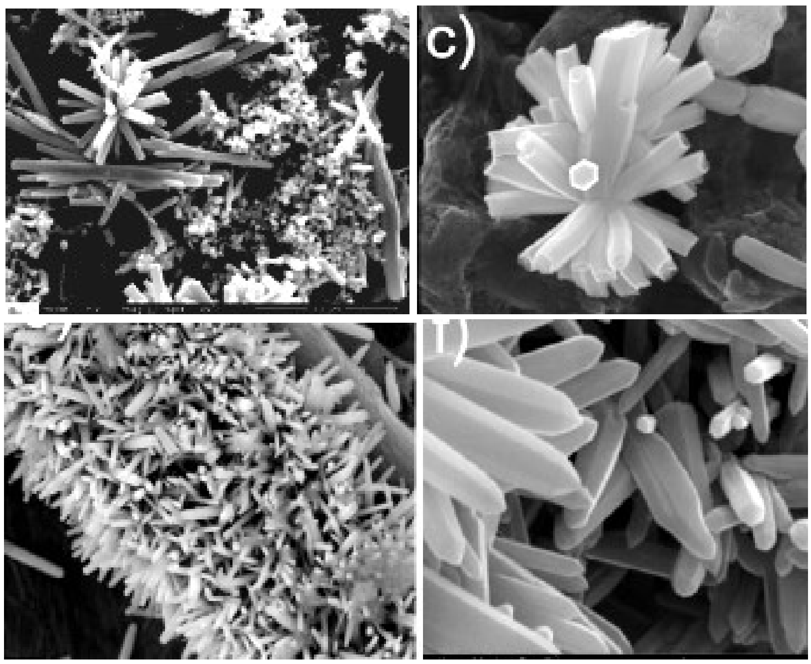


Fig. 9 Structure of ZnO microflower and nanorods grown on cellulosic substrate by hydrothermal process [25]

Paper [26] reports the synthesis and UV sensing characteristics of a cellulose and ZnO hybrid nanocomposite (CEZOHN) prepared by exploiting the synergetic effects of ZnO functionality and the renewability of cellulose. Vertically aligned ZnO nanorods were grown well on a flexible cellulose film by direct ZnO seeding and hydrothermal growing processes.

The ZnO nanorods have the wurtzite structure and an aspect ratio of 9 ~ 11. Photo-response of the prepared CEZOHN was evaluated by measuring photocurrent under UV illumination. CEZOHN shows bi-directional, linear and fast photo-response as a function of UV intensity.

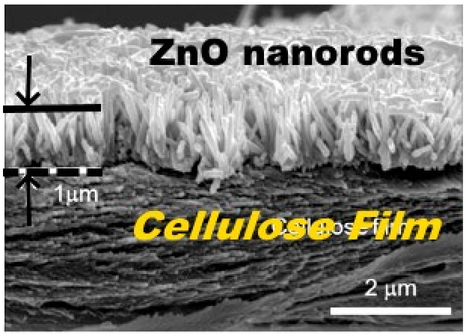
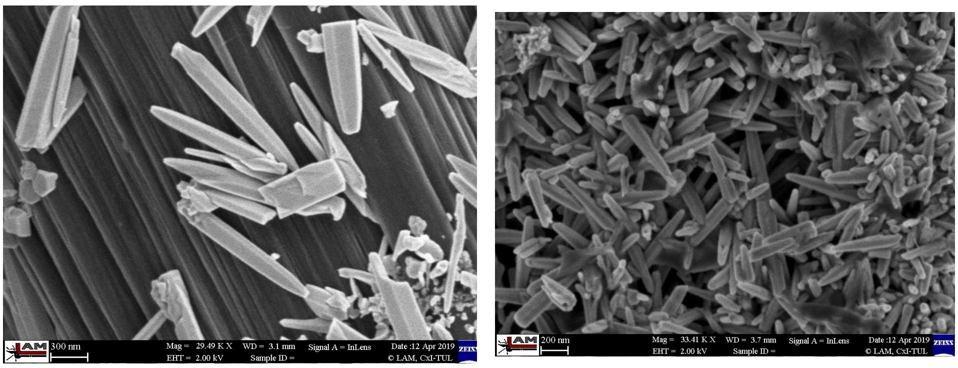


Fig. 10 Morphology of grown ZnO nanorods of cellulosic film [26]

Electrode materials, light sources, repeatability, durability and flexibility of the prepared CEZOHN were tested and the photocurrent generation mechanism is discussed.

The silver nanowire coating used for electrodes on CEZOHN is compatible with a transparent UV sensor. The prepared CEZOHN is flexible, transparent and biocompatible, and hence can be used for fexible and wearable UV sensors.

The whiskerization deals with growing a secondary reinforcement directly onto the surface of the fiber, and thereby enhanced interphase properties due to better cohesion between fiber and matrix. Therefore, the initial work is being carried out to study the effect of whiskerization on mechanical properties of laminated composites. The ZnO whiskers were grown on the surface of carbon fabrics using conventional and microwave hydrothermal method. Figure 10 shows the whiskerization of ZnO rods on the surface of carbon fabrics. The more uniform growth of ZnO rods was observed in case of microwave hydrothermal method than conventional hydrothermal method. Furthermore, the size of ZnO rods was found smaller for microwave hydrothermal method.



(a) Conventional hydrothermal method

(b) Microwave hydrothermal method

Fig. 11 Whiskerization of ZnO on the surface of carbon fabrics

ACKNOWLEDGEMENT

This work was supported by the Ministry of Education, Youth and Sports of the Czech Republic and the European Union - European Structural and Investment Funds in the frames of Operational Programme Research, Development and Education - project Hybrid Materials for Hierarchical Structures (HyHi, Reg. No. CZ.02.1.01/0.0/0.0/16_019/0000843) and within the project TH04020405 “Advanced Self-Sensing Materials for Critical Components of Rail Vehicles“, which is solved with the financial support of the Technology Agency of the Czech Republic (TAČR).

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