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ZnO nanowires decoration on carbon fiber via hydrothermal synthesis for paper-based friction materials with improved friction and wear properties

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ABSTRACT

Two-step growth of ZnO nanowires (NWs) on carbon fiber (CF) surface via hydrothermal synthesis was studied and their application in the preparation of paper-based friction materials by wet-forming process was also investigated. SEM and EDS results showed a dense and uniform ZnO NWs layer with vertical alignment was well established on surface of CFs. UV–vis spectra and XRD characterization further confirmed the formation of ZnO NWs on CFs surface. In comparison with control sample (paper-based friction material containing pristine CFs), the modified sample (paper-based friction material containing modified CFs) exhibited higher and more stable dynamic friction coefficient and greater wear resistance. It was concluded that the CFs@ZnO NWs had excellent tribological properties and was highly promising for wet paper-based friction material.

1. Introduction

Paper-based friction materials are widely used for oil-immersed clutches and brakes [1,2]. Friction materials should be endowed with stable friction coefficient, moderately low wear rate and good wear resistance under oil-lubricated conditions during the friction process [3,4]. Herein, the reinforced fibers play a critical role in maintaining the mechanical strength and excellent tribological properties of paper-based friction materials. Carbon fibers have been always used as reinforcements in composites for its high specific strength and modulus, good friction and wear performance, and excellent self-lubricating ability [5–7], which are essential for structural applications in friction materials. However, the smooth and chemically inert surface, and low chemical reactivity of the pristine CFs lead to the basal limitations of their applications in paper-based composites material because of poor interfacial interaction between fiber and matrix [8,9]. Therefore, strong adhesion at the interface is required to be established for the effective transfer throughout the interface and surface modification of CFs is of great importance for enhancing the interfacial adhesion, mechanical strength and tribology properties of paper-based composites.

In the past few decades, many strategies have been performed on the surface modification of CFs to improve interfacial adhesion of materials for broader industrial applications, like chemical and physical treatment [10–13], plasma treatment [14,15] and γ-ray irradiation [16]. Nevertheless, the chemical treatments lowered the strength of CFs to some extent because the fiber itself was damaged seriously. The plasma treatment required the specialized equipment, and γ-ray irradiation was limited in practice due to high-energy consumption and harsh reaction conditions. Recently, versatile nanostructures have drawn great interest to develop new composites with desirable properties [17]. Carbon nanotubes and graphene oxide were introduced onto the surface of carbon fibers by CVD in order to improve the interfacial properties and mechanical strength of carbon fiber/epoxy composites [18–21], but it was limited due to the fact that the degradation of carbon fiber when exposed to the CVD environment.

All these studies revealed that nanoparticles had not only attracted considerable attention due to its outstanding reinforcement potential, but also could significantly improve the comprehensive properties of resulting materials. For instance, a series of nanoparticles such as nanosilica, CuO nanowires, TiO_2 array, etc., have been utilized in friction materials to obtain excellent materials with outstanding friction and wear properties [22–25]. Zinc oxide nanowires (ZnO NWs), an environmentally friendly nanomaterial with large specific surface area, high surface energy and high thermal conductivity, have been studied

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in a variety of fields such as solar cells [26], gas sensor [27], light emitting diodes [28] and energy harvesting materials [29,30]. ZnO NWs have also been used as a whisker interphase to enhance structural properties of CFRP composites [31,32]. Many researches on ZnO NWs have been done for various advanced materials. However, there has been less study on the tribology properties with ZnO NWs embedded in friction materials, especially in the wet paper-based friction materials.

This study aims to demonstrate the growth of ZnO NWs on CFs via hydrothermal method and then used to prepare paper-based friction material by wet-forming process. SEM, EDS, UV and XRD these techniques were adopted to characterize the modified CFs. The key parameters of paper-based friction materials, including dynamic friction coefficient, wear rate and the worn surface morphologies were all evaluated and discussed. At the same time, the application of hydrothermal synthesis of ZnO NWs on surface of CFs in the paper-based composite materials will be expanded.

2. Experimental

2.1. Material

Zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$, 99.0%), sodium hydroxide (NaOH, 96%), ethanol (99.7%), Zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$, 99.0%), and hexamethylenetetramine (HMTA) ($C_6H_{12}N_4$, 99.0%) were supplied by Tianjin Damao Chemical Reagent Company in China and used without further purification. Dopamine hydrochloride (98%) and tris-(hydroxy-methyl) aminomethane (TRIS, 99%) were purchased from Chengdu Best Reagent Co. Ltd in China. Polyoxyethylene (PEO) ($Mw = 3 \times 10^6$–4×10^6) and cationic polyacrylamide (CPAM) ($Mw = 5 \times 10^6$–8×10^6) were from Sumitomo Refining Co., Ltd., Japan. Carbons fibers (average diameter of 6–8 μm and length of 3–5 mm) were produced by Nanjing Weida Composite Material Co., Ltd., China. Fillers, including sepiolite velvet, diatomite, alumina powder and the friction performance regulator such as graphite, were all used in the preparation of paper-based friction materials.

2.2. CFs modified with ZnO NWs via hydrothermal method

In the first functionalized step, carbon fiber was immersed in acetone for 24 h to remove the surface sizing and other impurities. Subsequently, the fibers were placed in a mixed solution of dopamine hydrochloride (2.0 g/L) and tris-buffer (1.2 g/L) solution with a pH of 8.5 [33]. Then, the polydopamine (PDA) coated CFs were obtained after 24 h reaction at room temperature with a thoroughly washing in deionized water for 3 times and drying at 80 °C for 2 h.

In the second modification step, the ZnO NWs were grown on surface of CFs via a two-step process consisting of hydrothermal synthesis and hydrothermal growth as described in the previous work [34]. The hydrothermal synthesis was the formation of ZnO seed coating layer on CFs. The PDA treated CFs were dipped in the ZnO seed solution which was prepared by mixing 200 mL ethanol solution containing 0.01 g zinc acetate dihydrate and 40 mL ethanol solution containing 0.04 g NaOH at 65 °C under constant mechanical stirring, then diluting to 400 mL by adding ethanol. The soaked CFs were annealed at 150 °C for 15 min for three times to strengthen the adhesion between the ZnO seed and the CFs. The hydrothermal growth of ZnO NWs on the CFs was conducted in the ZnO growth solution obtained by mixing zinc nitrate hexahydrate (0.025 M) and hexamethylenetetramine (HMTA) (0.025 M) in the deionized water. The growth was experimented in a sealed container at 95 °C for 4 h. The resulting CFs were collected by filtering and thoroughly washed with deionized water, and then dried at 105 °C for 12 h in vacuum oven.

2.3. Preparation of paper-based friction materials

Paper-based friction materials were prepared by wet-forming, impregnation and hot-press processes, as described previously [35]. The paper sheet was fabricated with a mixture of modified CFs, aramid fiber and other fibers and fillers in a hand sheet former (TD10-200, Xianyang, China) with the quantitative weighing of 100 g/m^2. The wet-forming process was conducted as follows. The fibers and fillers were mixed to form the pulp suspension. Subsequently, PEO used as dispersant and CPAM used as retention and drainage aid were added into the suspension as usual. After stirring well in a pulp disintegrator (L&W991509, Sweden), the pulp suspension was poured into the container of handsheet former to drain thoroughly to form a wet paper sheet until the pulp was separated into single fiber in water with stirring. The wet paper sheet was dried and then impregnated with phenolic resin using ethanol as solvent. After impregnation, the paper sheet was dried again in oven and hot-pressed by vulcanizing machine (Qingdao, China) at 150 °C for 2.5 min with pressure of 15 MPa, resulting in the paper-based friction material. The control samples of paper-based friction materials were prepared by using the same process as above.

2.4. Characterization and analysis

The morphology of modified CFs and the worn surfaces of paper-based friction material were observed by scanning electron microscope (SEM, VEGA-3-SBH, Czech). The samples were sputtered with gold and observed with secondary electron imaging mode and an operating voltage of 20 kV. Energy dispersive spectroscopy (EDS, Octane prime, U.S.A.) was used to detect ZnO NWs on the surface of CFs. Ultraviolet-visible spectrophotometer (UV–vis, Cary 5000, Agilent) was used to record the UV adsorption spectrums of modified CFs to confirm ZnO NWs on surface of CF. CFT-I multi-functional material surface performance tester (Lanzhou, China) was applied to test the tribological properties of the control sample and the modified sample under a load of 100 N and a speed of 300r/min. Additionally, the samples were required to be immersed in lubrication oil for 12 h before the friction and wear test.

3. Results and discussion

3.1. Concept of CFs@ZnO NWs preparation for improving their tribological properties in the preparation of friction materials

Fig. 1 illustrated the concept of ZnO NWs growth onto CFs surface by hydrothermal method. The pristine CFs (Fig. 1(a)) were pretreated by PDA (Fig. 1(b)) to obtain chemically active CFs, which could introduce some hydroxyl groups on its surface, as shown in Fig. 1(c). Fig. 1(d) showed the PDA treated CFs were dipped in ZnO seed solution to obtain a stable and homogeneous suspension. Then, ZnO crystal seed layer on surface of CF was produced through coordination and hydrogen bonding between ZnO and PDA treated CF (Fig. 1(e)). Fig. 1(f) showed the CFs with ZnO crystal seeds were immersed and dispersed in ZnO growth solution for the growth of ZnO NWs onto CFs surface through the process shown in Fig. 1(g), thus generating the final modified CFs (Fig. 1(h)).

The proposed concept was based on:

1) The zinc acetate dehydrate could react with sodium hydroxide to form zinc hydroxide ($Zn(OH)_2$), and zinc hydroxide and surplus hydroxide ion further reacted to form coordination ligand ($[Zn(OH)_4]^{2-}$) under strong alkaline conditions, thus producing ZnO crystal seeds layer by dissociation of the coordination ligand.
2) The HMTA in the ZnO growth solution could be decomposed into formaldehyde and ammonia when heated (Fig. 1(g)), then Zn^{2+} and the hydroxide ion could react to form zinc hydroxide again under strong alkaline conditions and further generating the ZnO NWs along with the ZnO crystal seeds.
3) Dopamine could be self-polymerized to form polydopamine on the