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Preparation, electrical conductivity, photocurrent and wettability of carbon microcoils

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Abstract. In this paper, we report on fabrication and physical properties of carbon microcoils, which are prepared by a chemical vapor deposition (CVD) process with Ni-catalyzed pyrolysis of acetylene, and characterized by a scanning electron microscope, a transmission electron microscope and an infrared spectrometer. The dark electrical conductivity of an isolated carbon microcoil is about 81 S/cm at room temperature, and its temperature dependence follows three-dimensional Mott variable-range hopping (VRH) model. Particularly, evident photocurrent is observed in the carbon microcoil upon cameral flash illumination. In addition, it is found that the surface of microcoil film is hydrophobic, showing a large water contact angle of about 135°. These results indicate that carbon microcoils have excellent physical properties, and can be used as optoelectronic and reinforced materials.

Introduction

Carbon micro- and nanocoils with unique three-dimensional (3D) structures have drawn much attention in the last decade due to their potential applications as high-performance electromagnetic wave absorbers [1-3], micro-/nanoscale solenoids [4,5], heaters [6] and springs [7], electromechanical sensors [8], tactile sensors [9], and a novel reinforcement in high-strain and semiconductive composites [10,11]. Carbon micro- and nanocoils can be prepared by different methods. For example, thermal chemical vapor deposition (CVD, e.g., Ni catalyzed pyrolysis of acetylene) [5], plasma enhanced CVD [12], and laser-assisted CVD [13]. Their morphology, structure characterization, growth mechanism [5,14], electromagnetic wave absorption properties [1-3], mechanical [7,8,15,16] and electrical [15,17] properties have also been extensively studied. For example, Zhao and Shen explored the microwave absorption properties of carbon nanocoils [3]. It was found that the reflection loss of the Nomex honeycomb sandwich composites filled with the carbon nanocoils was below -10 dB (90% absorption) at 8.9-18 GHz in the range of 2-18 GHz, and the minimum value was -32.23 dB at 12 GHz [3]. Carbon micro- and nanocoils show excellent mechanical properties [7,8,15,16]. It was reported that the nanocoil was monotonically loaded/unloaded in tension to a maximum coil extension of 33%, and behaved like an elastic spring with a spring constant of 0.12 N/m in the low strain region [7]. The shear modulus of the amorphous carbon microcoils was estimated to be about 3 GPa [16]. The mechanical and electrical properties of carbon nanocoils were also studied by a manipulator-equipped scanning electron microscope. The carbon nanocoils had an electrical conductivity of 100-180 S/cm and Young modulus of 0.1 TPa at room temperature [15]. Shen et al. [17] reported the low-temperature dc electrical conductivity and magnetoresistance of a single microcoiled carbon fiber. However, photoconductivity and wettability of carbon micro-/nanocoils have not intensively reported yet.

In this paper, carbon microcoils have been prepared by a thermal CVD process with Ni-catalyzed pyrolysis of acetylene, and characterized by electron microscopy. Particularly, dark electrical conductivity and photocurrent of an individual carbon microcoil have been studied. Furthermore, wettability (water contact angle) of a thin film made of carbon microcoils has also been explored.

Experimental details

The carbon microcoils were prepared by the Ni-catalyzed pyrolysis of acetylene, a conventional CVD method reported previous [5]. In a typical synthesis procedure, a ceramic boat with 0.06 g Ni powder (with an average diameter of 5 μm) was placed in the central part of a horizontal reaction quartz tube (16 mm inner diameter, 1.2 m length) which was heated in argon atmosphere. The reaction conditions were controlled as follows: temperature of 750-790 ˚C for 1 h, and argon, H_2, and C_2H_2 flow rates of 40, 120, and 30 ml/min, respectively. After cooling, black carbon microcoils could be obtained.

The resultant carbon microcoils were characterized by a scanning electron microscope (SEM; JEOL JSM-6390), a transmission electron microscope (TEM; JEOL 100CX) and an infrared (IR) spectrometer (MAGNA-IR 550). For the measurements of dark electrical conductivity and photoconductivity, an isolated carbon microcoil with a length of about 2.8 mm was selected and placed onto a Si/SiO_2 wafer, and then four gold wires with 20 μm in diameter were attached on the microcoil with highly conductive silver paste. The electrical conductivity in a wide temperature range from 300 down to 3 K, and the photocurrent upon camera flash illustration at room temperature were measured using a Keithley-220 current source and a Keithley-181 nanovoltmeter. The water contact angle values were acquired on a Dataphysics OCA-20 contact angle system at room temperature in ambient atmosphere. For each measurement, a 5 μL droplet was dispensed onto a compressed thin film made of carbon microcoils.

Results and discussion

Figure 1 shows the typical SEM and TEM images of the as-grown carbon microcoils. The regular coils generally have a regular 3D helical structure with a 0.1-0.3 μm thickness, a 2-8 μm coil diameter, and a 0.1-3 mm coil length. A carbon microcoil generally consists two tubules (Fig. 1b) which have the same pitch, twist with each other, because they grow in the same direction simultaneously from a diamond-shaped Ni seed [14,18]. Concerning the growth mechanism of carbon microcoils, it is usually attributed to an unequal carbon extrusion rate at different catalyst face [19]. Recently, it was found that impurity elements may dissolve into the metal catalyst and cause anisotropic carbon microcoil formation [14,20]. In addition, it was also reported that the substrate can alter the energy state of the metal catalyst and incur helical structure on carbon microcoil [21].

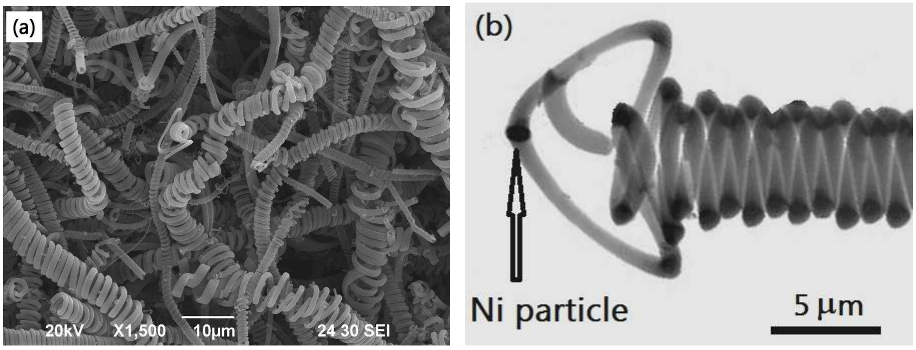

Figure 1 (a) Typical SEM and (b) TEM images of the as-grown carbon microcoils. A Ni particle as a catalyst on an individual carbon microcoil can be seen clearly.

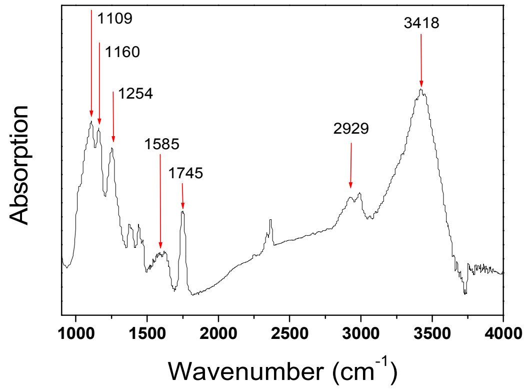

Figure 2 IR spectra of the as-grown carbon microcoils

Figure 2 shows the IR spectrum of the as-grown carbon microcoils. The broad absorption bands at about 1580-1620 cm^{-1} and about 1100-1300 (e.g., 1109, 1160 and 1254) cm^{-1} can be assigned to the aromatic C=C stretching vibration (graphitic structure) [22] with the bands of the C=O moieties and the C-O stretching vibration, respectively. The broad band at about 3418 cm^{-1} and the band at 1745 cm^{-1} can be assigned to the OH stretching vibration (absorbed water and/or carboxylic acid) and the C=O stretching vibration (lactone, carboxyl group, etc.) separately [23]. In addition, the band at about 2929 cm^{-1} can be attributed to C-H vibration of amorphous carbon deposited on the surface of coils [22,23]. Here, it is noted that the absorption band at about 1580-1620 cm^{-1} is broad and relatively weak, which is possibly due to the fact that the resultant carbon microcoils consist of the most part of an amorphous region with a small graphitizing region [22,23]. This point has been confirmed by the x-ray diffraction studies (data not shown).

In order to understand the electronic transport properties of the carbon microcoils, the electrical conductivity of an individual carbon microcoil was measured using a standard four-terminal method in a wide temperature range from 300 down to 3 K. The room-temperature conductivity is about 81 S/cm. For comparison, It has been reported that the conductivity of carbon nanocoils (196 nm in diameter and 1.5 μm long) is about 180 S/cm, which is smaller than a carbon nanotube, but larger than that of microcoils (~100 S/cm) consisting of amorphous carbon [15]. The differences are resulted from the crystallinity of the three kinds of materials.

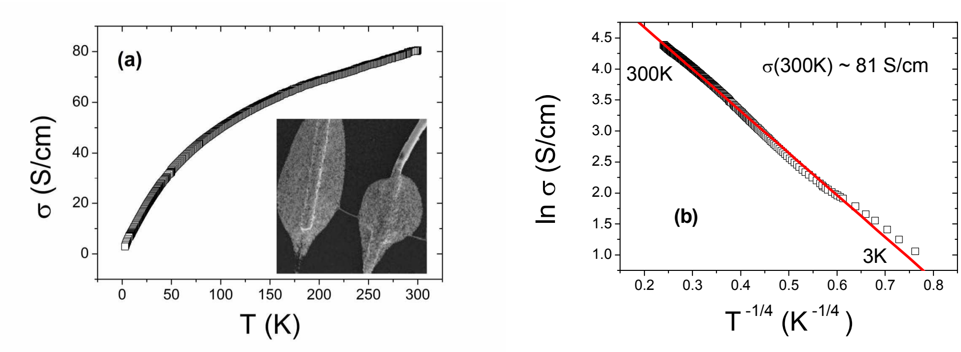

Figure 3 Temperature dependence of electrical conductivity of an individual carbon microcoil, plotted as (a) σ(T) vs T and (b) ln σ(T) vs $T^{-1/4}$. The inset shows an image of an isolated carbon microcoil with connected electrodes.

Figure 3(a) shows the temperature dependence of electrical conductivity of the carbon microcoil. It is obvious that the conductivity decreases with temperature lowing, exhibiting a semiconductor behavior. Further analysis indicates that the temperature dependent conductivity can be interpreted in terms of three-dimensional Mott variable range hopping (3D Mott-VRH) model [24], as shown in Fig. 3(b):

$$\sigma(T)=\sigma_0\exp[-(T_M/T)^{1/4}],$$

where σ_0 and T_M are parameters, $T_M = 18/L_c^3N(E_F)k_B$ is the characteristic Mott temperature, which can be determined from the ln $\sigma(T) \sim T^{1/4}$ plot, L_c is the localization length of charge carriers, k_B is the Boltzmann constant, and $N(E_F)$ is the density of states at the Fermi level. The Mott temperature, T_M, calculated from Fig. 3(b) is 2.1×10^3 K for the measured carbon microcoil. In fact, for amorphous carbon thin films [25], nanowires, and microcoils [17], a hopping-based (e.g., variable-range hopping) electron transport mechanism is usually cited as the dominant mechanism at temperature less than 250 K and thermally activated transport mechanism at higher temperatures [26].

Recently, the photoconductivity of single- and multi-walled carbon nanotubes has attracted extensive attention due to their potential application in micro- and macro-scale optoelectronic devices [27]. In this paper we also measured the photocurrent of an individual carbon microcoil upon cameral flash illumination at room temperature. The light intensity was about 6.25 mW/cm^2. As shown in Fig. 4, the current of the microcoil increases promptly from the dark current of 0.2~0.3 nA to about 0.9 nA upon flashing, and then decreases sharply to the dark current after flashing. This process could be well repeated. Similar results have been widely observed in single carbon nanotubes, carbon nanotube sheet and bundles [28-30]. The photocurrent could be ascribed to the photoinduced charge carriers (electron-hole pairs) generation in carbon microcoil and subsequent charge separation across the metal-carbon coil contacts [29].

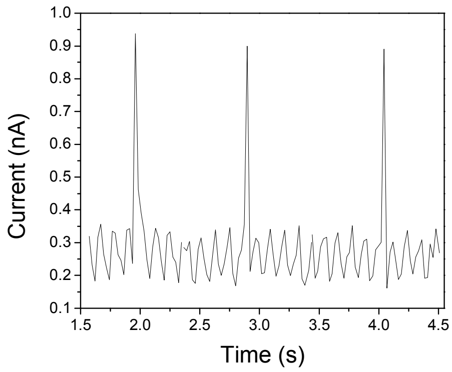

Figure 4 Photocurrent of an individual carbon microcoil generated upon camera flash illustration at room temperature

A static water contact angle (CA) is usually used as a criterion for the evaluation of hydrophilicity (CA<90°) or hydrophobicity (CA>90°) of a surface. Superhydrophobic surfaces with a CA larger than 150° have wide practical applications, such as self-cleaning surfaces of the production of textile, building glass, windshields of cars, etc. [31] There are many papers that reported wettability of carbon nanotubes, nanofibers, and composites [32], but only few papers reported water contact angles on surfaces of carbon micro-/nanocoils [33]. Compared with straight carbon fibers, highly coiled carbon coils have a higher surface area, where more air is trapped among them. According to Cassie and Baxter's theory [34], this porous rough surface is expected to have a special wettability: water becomes difficult to penetrate into the cavities of the surface, and the water droplets can easily slide/roll when the surface is tilted with a few degrees. Figure 5 shows an optical image of a water droplet on the thin film made of carbon microcoils. The contact angle is about 135±1°, which indicates that the surface is hydrophobic. Here it is noted that the water contact angle of carbon coils could be enhanced to larger than 150° through changing synthesis conditions [33].

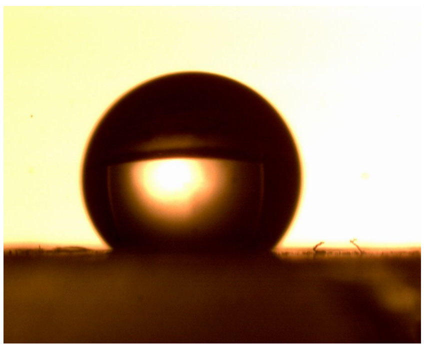

Figure 5 Water contact angle of thin film of carbon microcoils is about 135±1°.

Conclusion

Carbon microcoils have been successfully prepared by a CVD process with Ni-catalyzed pyrolysis of acetylene. The carbon coils generally have a regular 3D helical structure with a 2-8 μm coil diameter and a 0.1-3 mm length. The dark electrical conductivity of an individual carbon microcoil is about 81 S/cm at room temperature, and its temperature dependence follows 3D Mott VRH model. Evident photocurrent is observed in the carbon microcoil upon cameral flash illumination. In addition, a large water contact angle of about 135° is also observed on the surface of carbon microcoil film, indicating the film surface is hydrophobic. These results demonstrate that carbon microcoils have potential application in optoelectronic devices and reinforced materials.

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