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Soldering carbon nanotube fibers by targeted electrothermal-induced carbon deposition

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ABSTRACT

We introduce a facile approach to solder carbon nanotube (CNT) fibers by depositing carbon nanostructures at targeted fiber connections. Electrothermal induced deposition (ETID) process facilitates thermal chemical vapor deposition via current-induced Joule heating. Various carbon structures are formed between the overlapped fibers, with nanofibers covered by amorphous carbon and carbon nanowalls, resulting in effective soldering. The soldered connection between crossing fibers can be much stronger than the pristine CNT fiber: the separation force of the connection is measured to be up to 460 mN while the fracture force of the fiber is about 74 mN. The contact resistance decreased from >120 to 4.8 Ω after the ETID treatment. Such robust electrical soldering can be applied to connect CNT fibers in both parallel and cross configuration, to generate a soldered one-dimensional (1D) line, 2D network, and 3D cage.

1. Introduction

For everyday use of electrical energy, high performance electrical wires are of great importance. However, conventional metal-based wires might suffer from various problems including their high weight, skin effect, limited mechanical performance (especially in overhead power lines), and electromigration [1]. Furthermore, current carrying capacity (ampacity) has become another important factor for these conventional conductors [2,3]. Carbon nanotubes (CNTs) are considered as the most promising candidate of the next generation of wire, owing to their high ampacity (up to 10^9 A/cm) [4], tunable electronic structure (depending on tube chirality) [5], ballistic electron transport [6], high strength and modulus (up to 100 GPa and 1 TPa respectively) [7], light weight, and good corrosion resistance [8]. CNTs have been assembled into continuous fibers which can exhibit tensile strengths higher than 2 GPa and electrical conductivity up to 10^3–10^4 S/cm [1,9–12], and they are very promising candidates for the wire application, flexible sensors and other lightweight devices. But the problem of connecting CNT fibers hindered their development due to the inefficient electrical signals transfer between fibers.

Different from conventional metal-based wires, electron hopping between CNTs is the main mechanism that determines the fiber's electrical conduction behavior [13]. This means, once the fiber was broken, direct twisting two fiber ends together would not recover the conductivity due to the introduction of fiber-to-fiber contact resistance. To overcome this problem, deposition of a metal around CNT fiber could be a solution as it makes the fiber surface intimate to the metal [9,14]. In another promising method, soldering was attempted to join CNT fibers together with metal to reduce contact resistance [1]. Unfortunately, only a few metals (including Ti, Cr and Fe) have good wettability with CNT [15–18], making it difficult to widely apply the soldering method.

There are also other reports on soldering individual CNTs together or soldering CNTs to metal pads. For example, gold nanoparticle suspension was deposited on the area where CNT contacts the metal pad to obtain good ohmic or near-ohmic contacts [19]. Mechanical and electrical joining of crossing CNTs or a

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broken CNT was realized by depositing amorphous carbon via electron beam irradiation [20,21]. This method might be time-consuming and it is not trivial to macroscopic assembly of CNTs. But by following such deposition strategy, the formation of amorphous carbon should be an effective way to solder macroscopic CNT fibers.

In this paper, a novel and facile approach is developed to rapidly grow amorphous carbon (assembly of carbon nanofibers and nanowalls) at the contact between CNT fibers. These formed carbon nanostructures would joint CNT fibers, and the soldered structures have excellent mechanical and electrical performances. Based on this technique, simple and programmable circuits made of CNT fibers were built, demonstrating the simplicity of this soldering technique. Different kinds of CNT and graphene fibers can be soldered using the same technique by manually introducing iron catalysts into them. The approach developed here has potential to construct all-carbon-based circuits.

2. Experimental section

2.1. Synthesis of CNT fibers

The CNT fiber we used was fabricated by an injection chemical vapor deposition (iCVD), also called a floating catalyst CVD [22–26]. For the iCVD this growth, a mist of ethanol, ferrocene (2 wt%), and thiophene (1 vol%) was injected into a heated gas flow reactor (diameter 80 mm, temperature 1300 °C) under an injection rate of 20–30 mL/h. A gas mixture of argon (3500 sccm) and hydrogen (4250 sccm) were also injected into as a carrier gas. The grown CNTs were blown out by the carrier gas in a form of aerogel. The aerogel was led to pass through a water bath to get densified and become fiber-like. Finally, a continuous iCVD fiber with a length longer than several meters was collected on a winder [24,25]. Two kinds of fibers, with different diameters (d_f = 35–48/143–182 μm), were used in this study due to the change of growth parameters. The thick one was only used in the line-styled soldering. The CNT diameter ranged from 5 to 10 nm and there were rich double- and triple-walled CNTs [24].

2.2. Electrothermal soldering

The soldering system included a simple gas circuit, a bubbler, a reaction chamber, and a power system (a Zive BP2 system, WonATech Co. Ltd., Seoul, Korea). Argon gas was used as carrier gas to assist the injection of ethanol. Ethanol, the carbon source, was blown into the reaction chamber from the bubbler by the gas flow. The Ar gas flow was 100–200 sccm. The electric current was increased from zero to the designed value at a ramp rate of 1 mA/s. Other carbon sources, like acetone, acetic acid, etc., were tried and all of them were feasible.

2.3. Characterization

An optic measuring system (SKS-CL, Saike Digital Technology Exploiture, Co., Ltd., Shenzhen, China), a scanning electron microscope (SEM, Quanta 400 FEG, FEI, Hillsboro, USA) and a transmission electron microscope (TEM, Tecnai G2 F20S-TWIN, FEI, Hillsboro, USA) were used to characterize the sizes and morphologies of CNTs, the fiber and the formed nanostructures. The degree of the graphitization of these structures was measured by a Raman spectrometer (Labram HR, HORIBA Jobin Yvon, Paris, France). The change of iron content was recorded by EDX (APDLLO XL, Oxford instruments, Concord, USA). The electrical properties of all the samples were quickly tested by a multimeter (Fluke 15B, Fluke Co., Everett, USA) and further confirmed by a parameter analyzer (Keithley 4200A-SCS, Tektronix Inc., Beaverton, USA). A high-precision analytical balance (XP2U, Mettler-Toledo LLC., Columbus, USA) was used to measure the weight of the fiber. Mechanical properties of the untreated fibers and soldered fibers were carried out by a nano UTM testing system (T150, Keysight Technologies, Inc., Santa Rosa, USA) and an universal testing machine (Instron 3365, Instron, Norwood, USA), equipped with a 500-mN and a 10-N load sensor respectively.

3. Results and discussion

The new soldering strategy, namely electrothermal induced deposition (ETID), is a process involving local electrothermal heating of CNT fiber, thermal decomposition of a hydrocarbon and formation of carbon nanostructures. ETID was first applied to an unbroken CNT fiber. As reported by Wang et al. [26], the iCVD fiber was usually elliptical in its cross section. Therefore, an equivalent fiber diameter was calculated as d_f was used by considering the transfers and conjugate diameters ($2a$ and $2b$) of the ellipse, that is, $d_f = 2(ab)^{1/2}$. Here d_f was about 35–48 μm.

When tested in Ar atmosphere with a ramp rate of 1 mA/s, the fiber broke at I_{max} = 211 mA as a result of overheating, see the voltage-current (V–I) curve shown in Fig. 1a. It corresponded to an ampacity in an inert condition of 1.52×10^5 A/cm^2 (d_f = 42 μm). The fiber's resistance (R) increased with the current like most conductors and can be described by $R(T) = R_0[1+\alpha(T-T_0)]$, where R_0 is the resistance at T_0, mostly the ambient temperature [27]. As the temperature at fiber break could be much higher than 300 °C in air or Ar gas and up to 2000 °C in vacuum [28,29], ETID could take place around the fiber surface.

A typical ETID process was realized by placing a CNT fiber in an enclosed reaction chamber through which Ar gas and ethanol vapor were piped to flow. With increasing the electric current, the fiber temperature grew up rapidly and around a current threshold I_{growth}

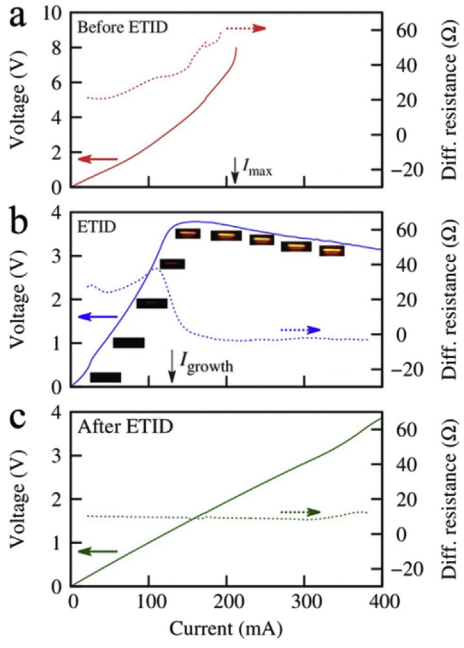

Fig. 1. V–I characterization of CNT fiber before (a), during (b), and after the ETID treatment (c). The electric current was increased by a rate of 1 mA/s. Differential resistance was calculated by $(V_{i+1}-V_i)/(I_{i+1}-I_i)$ where V_i and I_i are the averages in 20 s and V_{i+1} and I_{i+1} the averages in the following 20 s. The insets in (b) show the optical images of the fiber during the ETID. The fiber length was 8 mm. (A colour version of this figure can be viewed online.)