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Printing of highly conductive carbon nanotubes fibres from aqueous dispersion

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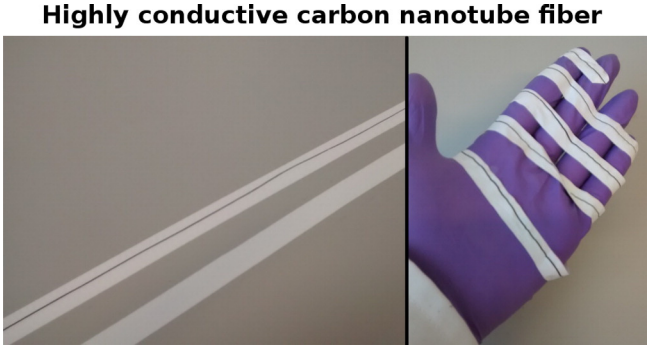
HIGHLIGHTS

- A method of printing carbon nanotubes (CNT) fibres from liquid suspension is presented.
- The resulting printed fibres are flexible, durable and of appreciable electrical properties.
- Electrical conductivity is enhanced by an order of magnitude as compared to CNT materials from other printing techniques.
- The proposed method of fibre production is straight-forward, scalable and can employ various CNT feeds.

GRAPHICAL ABSTRACT



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ABSTRACT

Carbon nanotubes (CNT) fibres were printed from liquid suspension. The resulting fibres are highly conductive, with conductivity close to the one of standard CNT fibres, flexible and very versatile. The printed fibres are comparable to fibres that can be gained from direct spinning, carpet spinning and spinning from superacids, but offer broader range of composition and are simpler and safer to produce as no high-temperature equipment and dangerous chemicals need to be used. Any CNT material can be implemented, allowing the use of a variety of CNT structures, different densities of the fibres and various shapes for the final product.

1. Introduction

CNTs have been the source of constant research in recent years. Throughout time, several different structures of CNTs themselves, as well as different assemblies of CNTs have been discovered and investigated. Starting from bundles of multi-walled nanotubes (MWNTs), followed by double- and single-walled nanotubes (DWNT and SWNT, respectively), later on individual CNTs as well as different assemblies have caught scientific attention [1–3]. Especially for technical application, macroscopic assemblies of CNTs seem to be advantageous as light weight and highly conductive wires can be obtained [4,5]. Amongst the most promising assemblies, CNT carpets [6,7], films [8,9] and fibres [10,11] have been thoroughly researched. Carpets are vertically aligned assemblies of CNTs, which are limited in length to the specific length of single CNTs [12–14]. Films and fibres on the other hand, which can either be spun from carpets [15] or produced in high temperature furnaces [16] amongst others, can be manufactured in

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theoretically unlimited length and width. Alignment of the CNTs within the films or fibres can be influenced by for example the speed of the direct-spinning [17]. This allows the production of light weight, highly conductive material that can compete with copper with respect to specific conductivity [18] and resistance to corrosion [19].

All methods used for spinning of highly conductive fibres have the drawback of being time- and cost-intensive, requiring highly specialised equipment and handling or involve hazardous chemicals. For instance, a popular wet-spinning method devised by Ericson et al. has the ability to print fibres, but they have to be suspended first in extremely dangerous superacid solutions [20,21]. As an alternative, one can produce the fibres by the direct-spinning method, but it requires high temperature furnaces and does not offer enough control over the inherent CNT structure yet [16]. Lastly, CNT macroassemblies can be spun from pre-synthesized CNT arrays [15,22], but not only often particular carpets are not spinnable, but also the process itself has a batch character. At the moment, there is no simple method of continuous production of CNT films or fibres from a desired type of CNTs.

The method of production of printed CNT fibres explained here starts with CNTs from either carpets or powders, providing an easy and fast way forward for the production of CNT fibres that can compete in performance with the priory described fibres from the spinning processes mentioned above. The use of CNT carpets and powdered CNTs also allows for fibres to be produced from all the different possibly available CNT structures and a fast adoption of new, for example highly sorted or conductive material, as soon as it becomes available. This makes production very straight forward. In a recently published paper by Huang et al. a similar approach was used to obtain CNT fibres from CNTs in aqueous dispersion [23]. Another report also showed the potential of inkjet printing of CNT dispersion [24]. Herein however, we report a production method of CNT fibres with orders of magnitude higher conductivity with easily accessible insulation, on which they are formed.

2. Experimental

2.1. Materials

Vertically-aligned CNTs (so-called CNT carpets) were grown by catalytic Chemical Vapor Deposition (cCVD) by a procedure reported elsewhere [25]. In brief, carbon source (toluene) and catalyst (ferrocene, 5.6 wt%) were continuously injected into a quartz tube placed inside a high-temperature furnace kept at 760 °C under argon atmosphere. As the synthesis progressed, CNTs were formed on the quartz tube walls, from which they were finally harvested upon the reaction completion after 8 h (recorded array height *ca* 0.7 mm). In this protocol, multi-wall (MWNT) CNTs (average diameter of 25 nm) are commonly formed. TEM micrograph of the material used for this study is shown in Fig. 1.

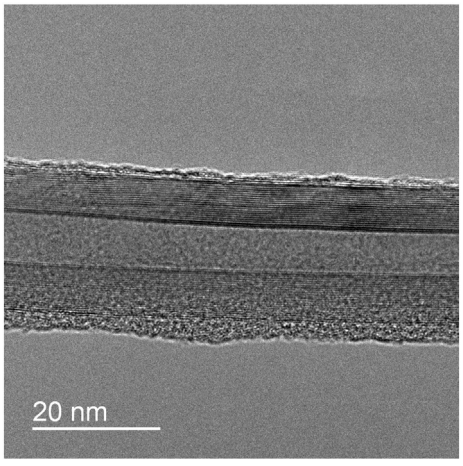

Fig. 1. TEM micrograph of the as-made CNT material.

2.2. Preparation of dispersion and printing

The CNT fibres were printed from CNT containing dispersions. The dispersions were prepared by sonication of CNTs using an in-house grown MWNT carpet and sodium dodecylbenzenesulfonate (SDBS) in distilled water. The material was sonicated with a tip-sonicator (Branson 450 CE with a 19 mm disruptor horn). In order to achieve a dispersion with a high enough content of CNT and a good stability, a high CNT loading was aimed for. A maximum of 2 wt% CNTs and 2 wt% SDBS could be solubilised at a time. A higher content of material would not homogeneously disperse, but stay in agglomerations. A lower content of CNTs and SDBS could have been solubilised, but due to a desired high loading of the dispersion, this was not found to be ideal. In order to minimise the damage to the CNTs by the sonication process, low power intensity (between 15 and 30 W) was chosen and sonication was performed over an extended period. An initial sonication at higher power (about 60 W) for an hour could be used to support the reduction of the initially big clusters of CNTs as present in the carpets and initiate further debundling of individual CNTs at lower sonication power. The exact time is correlated to the amount of material being sonicated at one time. Exemplary values are about 3 h for a 25 ml dispersion to up to 25 h for 150 ml of dispersion. The resulting dispersion was filtered through a metal sieve to separate non-dispersed carbon agglomerates from the dispersion. The top layer of non-dense dispersion was decanted, and the resulting dispersion was kept at 100 °C for a prolonged period of time to remove excess solvent and achieve a higher CNT loading. The resulting dispersion was dispensed through a hypodermic stainless steel needle (0.7 mm in diameter) onto a collecting spindle with the help of an electric motor (Fig. 2). Substrates of various widths can be employed. The amount of dispersion deposited was controlled by an infusion pump to which it was attached.

2.3. Characterization

TGA measurements were performed in the flow of air (20 ml/min) using Mettler Toledo TGA/DSC, with a heating rate of 10 °C/min on a temperature range from 25 to 900 °C.

Raman spectra were taken on an in-house build system with a 632.8 nm laser.

SEM and TEM were acquired on Nova NanoSEM (5 kV acceleration voltage) and FEI Tecnai Osiris (200 kV acceleration voltage), respectively.

2.4. Electrical testing

A two-point-probe measurement of electrical resistance was recalculated into the absolute value of electrical resistance by taking

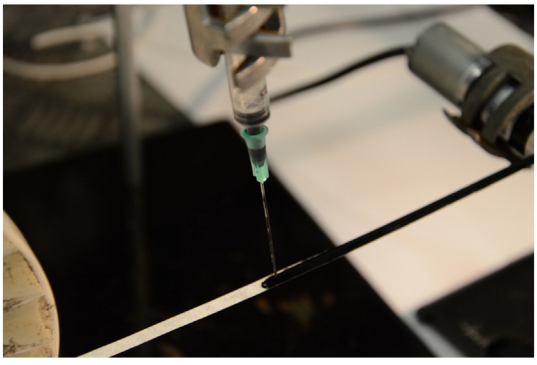

Fig. 2. Printing of CNT dispersion.