

**IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES,  
METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED  
THEREFROM**

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**BACKGROUND**

Lithium batteries are used extensively for portable electronic equipment and increasingly being used to provide electrical back-up for wind and solar energy. The commercially used Lithium ion (Li-ion) rechargeable batteries based on cobalt, nickel or manganese oxides for the cathode materials are known for their poor electrical conductivity and poor electrochemical stability which results in poor cycling (charge/discharge) ability. Olivine structures such as Lithium iron phosphate,  $\text{LiFePO}_4$  or lithium manganese phosphate,  $\text{LiMnPO}_4$ , operate at higher charge and discharge rates, but are still limited by low electrical conductivity and the kinetics of Li-ion diffusion.

Attempts to overcome the above deficiencies have included synthesis of nanocrystals of lithium salts as well as mixtures of lithium salts and carbon nanotubes. Even though the mixtures of carbon nanotubes and nanocrystals of lithium salts show improvements in charge/discharge rates the carbon nanotubes are not considered to be essentially discrete, i.e., individual tubes, nor attached to the tube. Also, the lithium nanocrystals are not ionically or covalently attached to the surface of the discrete carbon nanotubes to provide for intimate electron transfer and enhanced mechanical strength of the active material. Lithium salts have also been carbon coated to enhance electrical conductivity, but the carbon coating can slow the Lithium ion transport and may also react unfavorably with electrolytes over time.

In this invention we overcome the difficulties of low electrical conduction, improved lithium ion transport and mitigation of potentially damaging chemical side reactions by attaching nanosized crystals or nanosized layers of lithium ion active material to the surface of dispersed carbon nanotubes.

Likewise, for the lithium ion anode materials, active anode materials such as tin oxide or silicon can be attached to the discrete carbon nanotube surfaces to provide numerous benefits such as improved conductivity and mechanical strength.

In addition to the discrete carbon nanotube network providing support and spatial stabilization of nanosized particles or layers for cathode or anode material of the Lithium ion battery, other benefits include improved heat transfer media to avoid thermally-induced runaway, structural reinforcement to mitigate cracking of the active cathode or anode material due to expansion and contraction during charge and discharge cycles, and high surface area per weight of lithium ion active material to provide good energy density.

## DETAILED DESCRIPTION

### Experimental Examples

The following experimental examples are included to demonstrate particular aspects of the present disclosure. It should be appreciated by those of ordinary skill in the art that the methods described in the examples that follow merely represent illustrative embodiments of the disclosure. Those of ordinary skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments described and still obtain a like or similar result without departing from the spirit and scope of the present disclosure.

**Example 1.** Discrete Carbon Nanotubes with attached copper oxide nanoparticles suitable for a Lithium ion battery anode material.

102 mg of multi-wall carbon nanotubes oxidized to about 8 weight percent and aspect ratio about 60 are added to 100 mg copper sulfate, 640 mg sodium EDTA, 15 mg of polyethylene glycol, 568 mg of sodium sulfate and 60 mL of deionized water. The mixture is sonicated for 10 minutes and then heated to 40°C. 3 mL of formaldehyde (37% solution) and 500 mg of sodium hydroxide is added to bring the pH to 12.2. The mixture is stirred for 30 minutes at 85°C and then filtered using a 5 micron PVDF filter and washed with 200 mL of deionized water.

**Example 2.** Discrete Carbon Nanotubes with lithium iron phosphate coated on the surface of the tubes, suitable for a Lithium ion battery cathode material.

0.2 grams of multi-wall carbon nanotubes oxidized to about 8 weight percent and aspect ratio about 60 are added to 23.5 grams of diethylene glycol and sonicated for 15 minutes. The solution is grey. 1.16 grams of iron acetate dissolved in 4 ml of deionized water is added and the mixture

heated under nitrogen, stirring and sonication. After 1 hour at 140 degrees centigrade a solution of 0.65 grams of lithium acetate and 0.74 grams of ammonium phosphate in 4.2 ml of deionized water is added. The temperature is raised to about 185 degrees centigrade over 30 minutes then held at 185 degrees centigrade for 4 hours. The mixture is cooled to 110 C and sonicated for 5 minutes followed by further cooling to ambient temperature while stirring. The solids are separated from the mixture by centrifugation followed by ethanol washing. The resultant powder was dried in vacuo.

The resultant dried powder is determined to have a resistance of 2,000 ohm-cm compared to a commercially sample determined as 19,000 ohm-cm

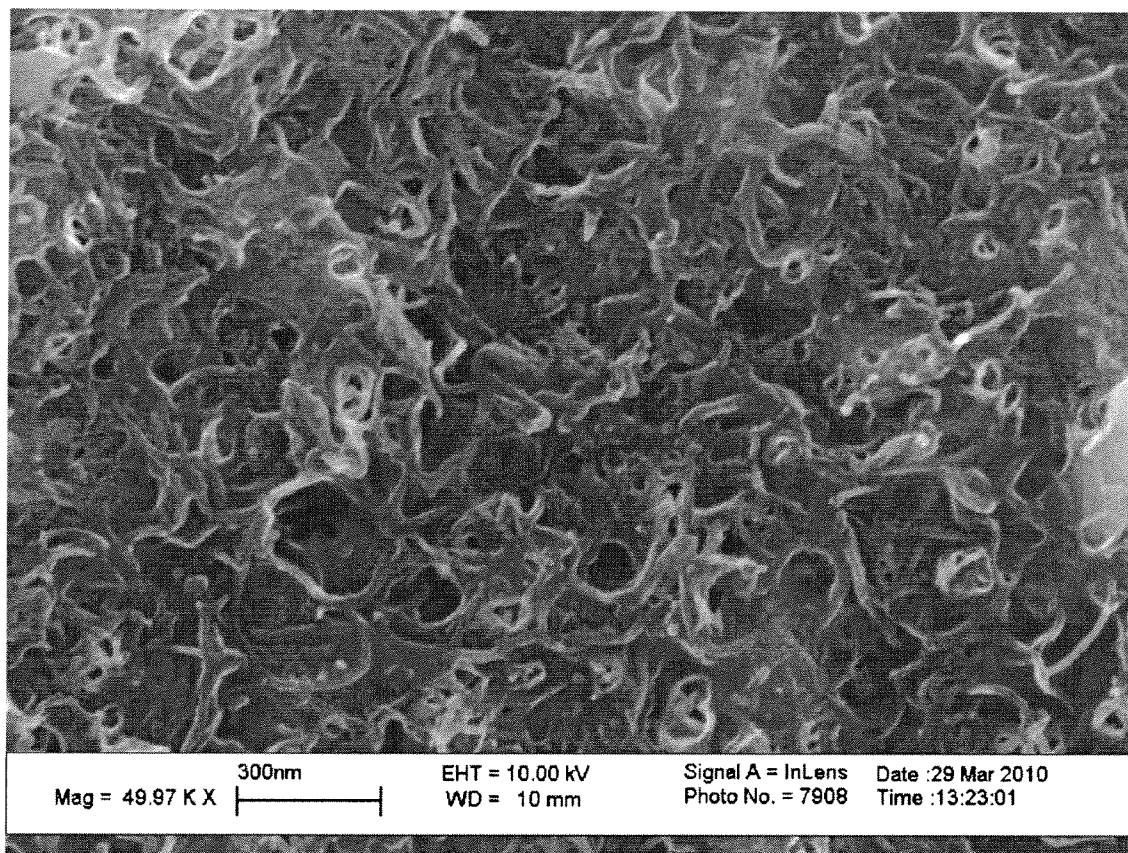
### Example 3.

Lithium iron phosphate obtained commercially is ground with multiwall carbon nanotubes oxidized to about 8 weight percent and of aspect ratio about 60, in a mortar and pestle at weight ratios of 5:1 and 1:1 lithium salt to carbon nanotube. Sodium dodecyl sulfate (SDS) is then added at a weight ratio of 0.7:1 compared to the carbon nanotube and deionized water added to resultant mixture to give a 0.5 weight percent solution of carbon nanotubes in the presence of lithium salt and surfactant. The mixture was sonicated for 1 hour, then the solids filtered using a PVDF filter (0.2 micron) washed with deionized water then dried. A micrograph of the discrete carbon nanotubes coated with lithium iron phosphate is shown in Figure 1. The results of the resistance of the powders is given in Table 1, units of resistance are ohm-cm.

Table 1

| Material               | 5:1 Weight ratio Lithium salt:carbon nanotube | 1:1 Weight ratio Lithium salt:carbon nanotube |
|------------------------|-----------------------------------------------|-----------------------------------------------|
| Dry mix                | 860                                           | 26                                            |
| Coated                 | 46.2                                          | 6.9                                           |
| Improvement of coating | 18.6 X                                        | 3.8X                                          |

Figure 1. Lithium salt coated as a nanolayer on discrete carbon nanotubes.



### CLAIMS

What is claimed is the following:

1. A composition useful for Lithium ion batteries comprising discrete carbon nanotubes having attached to their surface lithium ion active materials in the form of nanometer sized crystals or layers.
2. A composition of claim 1 wherein the lithium ion active material for a cathode comprises a lithium metal salt further comprising iron, manganese, cobalt, copper, nickel, vanadium, titanium, or mixtures thereof.
3. A composition of claim 2 further comprising the lithium metal salt with an olivine crystal structure.
4. A composition of claim 1 wherein the lithium ion active material for an anode comprises tin, silicon, copper, antimony, aluminum, Germanium or mixtures thereof.
5. A composition of claim 1 wherein the discrete carbon nanotubes have an aspect ratio of 25 to 500 and oxidation levels from 1 to 15% by weight of the carbon nanotube.
6. A composition of claim 1 further comprising graphene or oxygenated graphene.
7. A process for the production of the discrete carbon nanotubes having attached to their surface lithium ion active materials in the form of nanometer sized crystals or layers comprising the steps:
  - a) Selecting carbon nanotubes of aspect ratio 25 to 500 and oxidation level 1 to 15 weight percent.
  - b) Mixing the carbon nanotubes with a high boiling liquid, optionally with a non-ionic surfactant
  - c) Optionally sonicating the carbon nanotube liquid mixture
  - d) Adding reagents in correct balance to synthesize the desired lithium salt
  - e) Reacting the mixture at the desired temperature to form the lithium salt while sonicating the mixture
  - f) Separating the solid salts from the liquid.
  - g) Drying and annealing under an inert atmosphere the solid discrete carbon nanotubes with attached lithium salts at sufficient temperature to obtain the olivine crystal structure.
8. A process for the production of the discrete carbon nanotubes having attached to their

surface lithium ion active materials in the form of nanometer sized crystals or layers comprising the steps:

- h) Selecting carbon nanotubes of aspect ratio 25 to 500 and oxidation level 1 to 15 weight percent.
- i) Mixing the carbon nanotubes and desired lithium salt in a liquid with surfactant,
- j) Sonicating the carbon nanotube/lithium salt liquid mixture,
- k) Separating the solid salts from the liquid,
- l) Drying and annealing under an inert atmosphere the solid discrete carbon nanotubes with attached lithium salts at sufficient temperature to obtain the olivine crystal structure.

**ABSTRACT**

In various embodiments, exfoliated carbon nanotubes are described in the present disclosure. The carbon nanotubes maintain their exfoliated state, even when not dispersed in a medium such as a polymer or a liquid solution. Methods for making the exfoliated carbon nanotubes include suspending carbon nanotubes in a solution containing a nanocrystalline material, precipitating exfoliated carbon nanotubes from the solution and isolating the exfoliated carbon nanotubes. Nanocrystalline materials may include nanorods, hydroxyapatite and various hydroxyapatite derivatives. In some embodiments, methods for making exfoliated carbon nanotubes include preparing a solution of carbon nanotubes in an acid and filtering the solution through a filter to collect exfoliated carbon nanotubes on the filter. In some embodiments, a concentration of carbon nanotubes in the acid is below the percolation threshold. In other various embodiments, energy storage devices and polymer composites containing exfoliated carbon nanotubes are described herein. The energy storage device may be a battery containing at least two electrodes and an electrolyte in contact with the at least two electrodes. At least one of the electrodes in the energy storage device advantageously contains exfoliated carbon nanotubes. The polymer composites are prepared by mixing exfoliated carbon nanotubes with a polymer material. After being mixed in the polymer material, the carbon nanotubes maintain their exfoliated state.

| Provisional Application for Patent Cover Sheet                                                                                              |             |                                                                                                                |                                               |       |                                       |
|---------------------------------------------------------------------------------------------------------------------------------------------|-------------|----------------------------------------------------------------------------------------------------------------|-----------------------------------------------|-------|---------------------------------------|
| This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c)                                                      |             |                                                                                                                |                                               |       |                                       |
| <b>Inventor(s)</b>                                                                                                                          |             |                                                                                                                |                                               |       |                                       |
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| Given Name                                                                                                                                  | Middle Name | Family Name                                                                                                    | City                                          | State | Country ;                             |
| Clive                                                                                                                                       | P.          | Bosnyak                                                                                                        | Dripping Springs                              | TX    | US                                    |
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| <b>Title of Invention</b>                                                                                                                   |             | IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS... |                                               |       |                                       |
| Attorney Docket Number (if applicable)                                                                                                      |             | DENA-0008 (137124.00020)                                                                                       |                                               |       |                                       |
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**Entity Status**

Applicant claims small entity status under 37 CFR 1.27

☒ Yes, applicant qualifies for small entity status under 37 CFR 1.27☐ No**Warning**

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| Signature  | //Amy U. Dunstan// |           |         | Date (YYYY-MM-DD)                       | 2011-06-23 |
| First Name | Amy U.             | Last Name | Dunstan | Registration Number<br>(If appropriate) | 61069      |

This collection of information is required by 37 CFR 1.51. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **This form can only be used when in conjunction with EFS-Web. If this form is mailed to the USPTO, it may cause delays in handling the provisional application.**

| Electronic Patent Application Fee Transmittal |                 |                                                                                                                                |               |                             |
|-----------------------------------------------|-----------------|--------------------------------------------------------------------------------------------------------------------------------|---------------|-----------------------------|
| <b>Application Number:</b>                    |                 |                                                                                                                                |               |                             |
| <b>Filing Date:</b>                           |                 |                                                                                                                                |               |                             |
| <b>Title of Invention:</b>                    |                 | IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM |               |                             |
| <b>First Named Inventor/Applicant Name:</b>   |                 | Clive P. Bosnyak                                                                                                               |               |                             |
| <b>Filer:</b>                                 |                 | Amy L. Ulfers/Debbie Sams                                                                                                      |               |                             |
| <b>Attorney Docket Number:</b>                |                 | DENA-0008 (137124.00020)                                                                                                       |               |                             |
| Filed as Small Entity                         |                 |                                                                                                                                |               |                             |
| <b>Provisional Filing Fees</b>                |                 |                                                                                                                                |               |                             |
| <b>Description</b>                            | <b>Fee Code</b> | <b>Quantity</b>                                                                                                                | <b>Amount</b> | <b>Sub-Total in USD(\$)</b> |
| <b>Basic Filing:</b>                          |                 |                                                                                                                                |               |                             |
| Provisional Application filing fee            | 2005            | 1                                                                                                                              | 110           | 110                         |
| <b>Pages:</b>                                 |                 |                                                                                                                                |               |                             |
| <b>Claims:</b>                                |                 |                                                                                                                                |               |                             |
| <b>Miscellaneous-Filing:</b>                  |                 |                                                                                                                                |               |                             |
| <b>Petition:</b>                              |                 |                                                                                                                                |               |                             |
| <b>Patent-Appeals-and-Interference:</b>       |                 |                                                                                                                                |               |                             |
| <b>Post-Allowance-and-Post-Issuance:</b>      |                 |                                                                                                                                |               |                             |
| <b>Extension-of-Time:</b>                     |                 |                                                                                                                                |               |                             |

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| <b>Miscellaneous:</b>    |          |          |        |                      |
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| Electronic Acknowledgement Receipt          |                                                                                                                                |
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| <b>EFS ID:</b>                              | 10376992                                                                                                                       |
| <b>Application Number:</b>                  | 61500560                                                                                                                       |
| <b>International Application Number:</b>    |                                                                                                                                |
| <b>Confirmation Number:</b>                 | 8442                                                                                                                           |
| <b>Title of Invention:</b>                  | IMPROVED LITHIUM ION BATTERIES USING DISCRETE CARBON NANOTUBES, METHODS FOR PRODUCTION THEREOF AND PRODUCTS OBTAINED THEREFROM |
| <b>First Named Inventor/Applicant Name:</b> | Clive P. Bosnyak                                                                                                               |
| <b>Customer Number:</b>                     | 25555                                                                                                                          |
| <b>Filer:</b>                               | Amy L. Ulfers/Debbie Sams                                                                                                      |
| <b>Filer Authorized By:</b>                 | Amy L. Ulfers                                                                                                                  |
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| <b>Receipt Date:</b>                        | 23-JUN-2011                                                                                                                    |
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| <b>Warnings:</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |                                |                           |                                                         |    |   |
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| <b>Warnings:</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |                                |                           |                                                         |    |   |
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| <b>Warnings:</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |                                |                           |                                                         |    |   |
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| 4                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | Provisional Cover Sheet (SB16) | PRO_Cover_Sheet.pdf       | 28841<br>4bd671c7c3c242b84f43b841c3095141c7<br>b8ab7    | no | 2 |
| <b>Warnings:</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |                                |                           |                                                         |    |   |
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| 5                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | Fee Worksheet (SB06)           | fee-info.pdf              | 29935<br>c0ed8998afc944ddb72daf3cb80ca8ldcc<br>3e419    | no | 2 |
| <b>Warnings:</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |                                |                           |                                                         |    |   |
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