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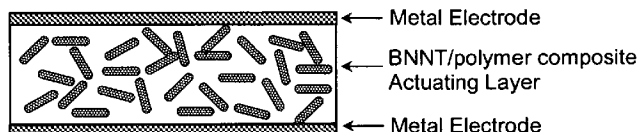
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(54) Title: ENERGY CONVERSION MATERIALS FABRICATED WITH BORON NITRIDE NANOTUBES (BNNTS) AND BNNT POLYMER COMPOSITES

FIG. 1A



(57) Abstract: Electroactive actuation characteristics of novel BNNT based materials are described. Several series of BNNT based electroactive materials including BNNT/polyimide composites and BNNT films are prepared. The BNNT based electroactive materials show high piezoelectric coefficients, d_{33} , about 14.80 pm/V as well as high electrostrictive coefficients, M_{13} , 3.21×10^{-16} pm²/V². The BNNT based electroactive materials will be used for novel electromechanical energy conversion devices.

**Energy Conversion Materials Fabricated with Boron Nitride Nanotubes
(BNNTs) and BNNT Polymer Composites**

**STATEMENT REGARDING FEDERALLY SPONSORED
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[01] The U. S. Government has a paid-up license in this invention and the right in limited circumstances to require the patent owner to license others on reasonable terms as provided for by the terms of Cooperative Agreement No. NCC-1-02043 awarded by the National Aeronautics and Space Administration.

CROSS REFERENCE TO RELATED APPLICATION

[02] The present application claims priority from U.S. Provisional Application Ser. No. 61/278,866, filed October 13, 2009.

BACKGROUND OF THE INVENTION

1. Field of the Invention

[03] The present invention relates to high performance energy conversion devices such as sensors and electromechanical actuators, and, more particularly to energy conversion devices manufactured from boron nitride nanotubes and BNNT/polyimide composite materials.

2. Description of Related Art

[04] Electroactive materials have been studied extensively in the last few decades for use in a variety of applications including electromechanical sensors and actuators, ultrasonic transducers, loudspeakers, sonars, medical devices, prosthetics, artificial muscles, electric energy harvesters and devices for vibration and noise control. Electroactive ceramics such as lead zirconate titanates (PZT), lead-lanthanum zirconate titanate (PLZT), and niobium-lead zirconate titanate (PNZT) have very high piezoelectric coefficients, but have poor mechanical properties (i.e., are brittle) and high toxicity. Compared to the electroactive ceramics, electroactive polymers such as poly(vinylidene fluoride) (PVDF) offer a unique combination of favorable characteristics because they are lightweight, conformable, and tough. However, they have relatively low electroactive coefficients and poor thermal properties.

[05] Recently, a series of amorphous piezoelectric polyimides containing polar functional groups have been developed, using molecular design and computational chemistry, for potential use as sensors in high temperature applications. The piezoelectric response of these polyimides is, however, an order of magnitude smaller than that of poly(vinylidene fluoride) (PVDF). This is due to the fact that the dipoles in the polymer do not align along the applied electric field efficiently because of limited chain mobility within the imidized closed ring structure. To increase the piezoelectric response of these polymers, synthesis with various monomers, control of the poling process, and the adding of carbon nanotubes (CNTs) have been reported.

[06] However, there are still limitations to the use of electroactive polyimide composites in many applications. For example, CNT doped polyimides have large leakage current because the CNTs are either conductors or narrow band gap semiconductors. This limits the use of the composites for high voltage devices. Furthermore, CNTs are chemically active and can be easily oxidized at elevated temperatures (above about 350°C in air).

[07] Novel electroactive materials have been required for increasing electroactive performance while reducing power consumption for many applications including in the aerospace field. Many electroactive materials have been proposed, but they still have problems of poor mechanical/thermal properties or unsatisfactory electroactive performance. Recently, boron nitride nanotubes (BNNTs) have been successfully synthesized, which exhibit excellent mechanical, electronic, optical, and thermal properties. BNNTs are thought to possess high strength-to-weight ratio, high temperature resistance (about 800°C in air), and radiation shielding capabilities. Furthermore, intrinsic piezoelectricity of BNNTs has been predicted theoretically. However, no experimental result of the piezoelectric properties of BNNTs or BNNT composites has been reported as yet. In this invention, we demonstrate electroactive actuation characteristics of novel BNNT based materials. We prepared several series of BNNT based electroactive materials including BNNT/polyimide composites and BNNT films. The BNNT based electroactive materials showed high piezoelectric coefficients, d_{13} , about 14.80 pm/V as well as high electrostrictive coefficients, M_{13} , 3.21×10^{-16} pm²/V². It is anticipated that the BNNT based electroactive materials will be used for novel electromechanical energy conversion devices.

[08] An object of the present invention is to provide high performance energy conversion devices.

[09] An object of the present invention is to provide high performance energy conversion devices such as sensors.

[10] Another object of the present invention is to provide high performance energy conversion devices such as electromechanical actuators.

[11] Yet another object of the present invention is to provide high performance energy conversion devices manufactured from boron nitride nanotubes and BNNT/polyimide composite materials.

[12] Finally, it is an object of the present invention to accomplish the foregoing objectives in a simple and cost effective manner.

SUMMARY OF THE INVENTION

[13] The present invention addresses these needs by providing a method for forming a boron nitride nanotube nanocomposite film, including the steps of combining a boron nitride nanotube solution with a polymer or ceramic matrix to form a boron nitride nanotube/polyimide mixture and synthesizing a boron nitride nanotube/polyimide nanocomposite film as an electroactive layer. The matrix is preferably synthesized from a diamine, 2,6-bis(3-aminophenoxy) benzonitrile ((β -CN)APB) and a dianhydride, pyromellitic dianhydride (PMDA). Alternatively, the matrix is polyvinylidenefluoride, polyvinylidenefluoride copolymer, polycarbonate or epoxy. The matrix can also be a highly elastic polymer such as polyurethane or

polysiloxane or a ceramic such as silicon dioxides or aluminum oxides. The the concentration of boron nitride nanotubes in the boron nitride nanotube/polyimide mixture is between 0 and 100% by weight. In an additional step, the boron nitride nanotube/polyimide nanocomposite film is coated with metal electrodes formed from chrome, gold or a mixture thereof. Alternatively, the boron nitride nanotube/polyimide film is coated with compliant electrodes formed from carbon nanotubes, carbon nanotube sheet, carbon nanotube/polymer composites, gold particles, silver particles or a mixture thereof.

[14] In one embodiment, a method for forming a boron nitride nanotube/polymer nanocomposite film, includes synthesizing a high temperature piezoelectric polyimide, combining a boron nitride nanotubes solution with the high temperature piezoelectric polyimide, using a polymer as a matrix and synthesizing a boron nitride nanotube/polyimide nanocomposite film as an electroactive layer. The polymer is dianhydride, pyromellitic dianhydride and the high temperature piezoelectric polyimide is synthesized from a diamine, 2,6-bis(3-aminophenoxy) benzonitrile ((β -CN)APB) and a dianhydride, pyromellitic dianhydride (PMDA). The concentration of boron nitride nanotubes in the boron nitride nanotube/polyimide mixture is between 0 and 100% by weight. In an additional step, the boron nitride nanotube/polyimide nanocomposite film is coated with metal electrodes, preferably formed from chrome, gold or a mixture thereof. Alternatively, the boron nitride nanotube/polyimide film is coated with compliant electrodes formed from carbon nanotubes, carbon nanotube sheeting, carbon nanotube/polymer composites, gold particles, silver particles or a mixture thereof.

BRIEF DESCRIPTION OF THE DRAWINGS

[15] A more complete description of the subject matter of the present invention and the advantages thereof, can be achieved by reference to the following detailed description by which reference is made to the accompanying drawings in which:

[16] Figure 1a shows a schematic diagram of a metal electroded BNNT/polymer composite actuator;

[17] Figure 1b shows a Schematic diagram of a carbon nanotube electroded BNNT actuator;

[18] Figure 2a shows a graph of thermally stimulated current (TSC) spectra of pristine polyimide and 2wt% BNNT/polyimide composite;

[19] Figure 2b shows a graph of remanent polarization (P_r) of pristine polyimide and 2wt% BNNT/polyimide composite;

[20] Figure 3 shows a proto-type BNNT actuator fabricated with carbon nanotube electrodes;

[21] Figure 4 shows a cross-sectional SEM image of a prototype BNNT actuator fabricated with carbon nanotube electrodes;

[22] Figure 5a shows a graph of the electric field induced strain of the BNNT actuator fabricated with CNT electrodes;

[23] Figure 5b shows a graph of the piezoelectric response of the BNNT actuator fabricated with CNT electrodes; and

[24] Figure 5c shows a graph of the electrostrictive response of the BNNT actuator fabricated with CNT electrodes.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

[25] The following detailed description is of the best presently contemplated mode of carrying out the invention. This description is not to be taken in a limiting sense, but is made merely for the purpose of illustrating general principles of embodiments of the invention.

[26] Since the first theoretical prediction of the existence of boron nitride nanotubes (BNNTs) in 1994 and the first experimentally synthesized BNNT report by Zettl's group in 1995, several types of BNNT synthesis methods have been reported. Recently, a new and conceptually simple method of producing extraordinarily long, highly crystalline BNNTs was demonstrated. BNNTs are thought to possess high strength-to-weight ratio, high thermal stability (up to about 800°C in air), piezoelectricity, and radiation shielding capabilities. Nakhmanson's theoretical analysis predicted that the piezoelectric coefficient of BNNTs can be higher than that of poly(vinylidene fluoride) (PVDF) or poly(vinylidene fluoride-trifluoroethylene) P(VDF-TrFE). However, the piezoelectric properties of BNNTs or BNNT composites have not been reported experimentally as yet. In this invention, we make use of the electroactive characteristics of novel BNNT based materials.

[27] First, a BNNT/polyimide nanocomposite film was synthesized as an electroactive layer by *in-situ* polymerization under simultaneous shear and sonication. The high temperature piezoelectric polyimide, used as a matrix for this invention, was synthesized from a diamine, 2,6-bis(3-aminophenoxy) benzonitrile ((β -CN)APB), and a dianhydride, pyromellitic dianhydride (PMDA). The concentrations of BNNTs in the polyimide were 0 and 2 wt%. In order to characterize electroactive properties of the composites, the samples were coated with metal (chrome/gold) electrodes for both sides (FIG 1a).

[28] Thermally stimulated current (TSC) spectra of the BNNT nanocomposites were obtained using a Setaram TSC II. Each sample was polarized by a direct current (DC) electric field of 5 MV/m at an elevated temperature ($T_p = T_g - 5^\circ\text{C}$) for a selected poling time ($t_p = 30$ min). The glass transition temperatures (T_g) of the pristine polyimide and 2% BNNT/polyimide composite, measured by a differential scanning calorimeter (DSC), are 274.3 and 271.4°C, respectively. After poling, the depolarization current was measured as the sample was heated through its glass transition temperature (T_g) at a heating rate of 7.0°C/min. As shown in Figure 2a, the pristine polyimide showed negligible depolarization currents until about 225°C, which indicates a good thermal stability of polarization, and then exhibited a rapid depolarization current with a maximum peak of 0.012 mA/m² at 255.9°C. On the other hand, the 2wt% BNNT/polyimide nanocomposite exhibited two depolarization peaks at 119.3°C and 255.5°C. The magnitude of the depolarization current of the nanocomposite was significantly larger than that of the pristine polyimide as seen in FIG 2b, and reached a maximum value of about 0.05 mA/m², five times greater than that of the pristine polyimide. The remanent polarization (P_r) was

calculated by integrating the current with respect to time and is plotted as a function of temperature as shown in FIG 2b. P_r is given by,

$$P_r = \frac{q}{A} = \frac{1}{A} \int I(t) dt \quad (1)$$

where q is the charge, A is the electrode area, I is the current, and t is the time. Details of conventional poling procedures have been described elsewhere [J. H. Kang et al., *NANO*, **1**, 77 (2006)]. The remanent polarization (P_r) of the 2wt% BNNT/polyimide nanocomposite was 12.20 mC/m², almost an order of magnitude higher than that of the pristine polyimide (1.87 mC/m²). In general, the piezoelectricity of a material is proportional to its remanent polarization. From the TSC result, adding BNNT, even only 2wt%, was proven to increase the piezoelectricity (remanent polarization) of the polyimide significantly.

[29] An all nanotube film actuator, with a BNNT active layer, was fabricated by a filtering method [J. H. Kang et al., *J. Polym. Sci. B: Polym Phys.* **46**, 2532 (2008)]. Single wall carbon nanotubes (SWCNTs) were used as electrodes for the actuator instead of metal. First, solutions of SWCNTs and BNNTs were prepared in N-methylpyrrolidone (NMP) under sonication. An adequate amount of the SWCNT solution was filtered through the surface of an anodized alumina membrane (pore size: 0.2 μm) to form a SWCNT film on the membrane. Then, the BNNT solution and finally the SWCNT solution were sequentially filtered onto the

SWCNTs film on the membrane to make a three layered (SWCNT/BNNT/SWCNT) “*all-nanotube actuator*” structure shown in FIG 3. The freestanding *all-nanotube actuator* film, shown in FIG 3, was easily delaminated by breaking the brittle membrane. To increase durability, polyurethane resin was infused into the all-nanotube actuator. FIG 4 shows the cross-sectional scanning electron microscopy (SEM) image of a prototype BNNT actuator fabricated with SWCNT electrodes (Hitachi S-5200 Field Emission Scanning Electron Microscope). The top and bottom layers are SWCNT electrodes and the middle layer is the BNNT actuating layer.

[30] In-plane strain (S_{13}) was measured using a fiber optic device while the sample was under an alternating current (AC) electric field of 1 Hz. The strain (S_{13}) of the sample appears as a superposed curve (black solid squares in FIG 5a) of linear and nonlinear strains as a function of frequency. The superposed curve was de-convoluted to a linear response (red solid circles in FIG 5a) and a nonlinear response (blue solid triangles in FIG 5a). The linear response seems to originate from the piezoelectric property of the BNNT active layer. From linear fitting of the data (FIG 5b), the piezoelectric coefficient, d_{13} was calculated to be about 14.80 pm/V. This is comparable to the values of commercially available piezoelectric polymers such as poly(vinylidene fluoride) (PVDF). The nonlinear response showed a quadratic increase with increasing applied electric field, indicating that the mechanism of this strain is mainly an electrostrictive response (FIG 5c). The electrostrictive coefficient (M_{13}) of the BNNT active layer, calculated from the slope of a plot of the strain (S_{13}) to the square of electric field strength (E^2), $S_{13} = M_{13} E^2$, was $3.21 \times 10^{-16} \text{ pm}^2/\text{V}^2$ on average. This value is several orders of magnitude higher than those of electrostrictive polyurethanes (-4.6×10^{-18} to $-7.5 \times 10^{-17} \text{ m}^2/\text{V}^2$).

[31] .Obviously, many modifications may be made without departing from the basic spirit of the present invention. Accordingly, it will be appreciated by those skilled in the art that within the scope of the appended claims, the invention may be practiced other than has been specifically described herein. Many improvements, modifications, and additions will be apparent to the skilled artisan without departing from the spirit and scope of the present invention as described herein and defined in the following claims.

What is claimed is:

1. A method for forming a boron nitride nanotube nanocomposite film, comprising:
combining a boron nitride nanotube solution with a matrix selected from the group consisting of polymer and ceramic to form a boron nitride nanotube/polyimide mixture;
synthesizing a boron nitride nanotube/polyimide nanocomposite film as an electroactive layer.
2. The method of claim 1 wherein the matrix is synthesized from a diamine, 2,6-bis(3-aminophenoxy) benzonitrile ((β -CN)APB) and a dianhydride, pyromellitic dianhydride (PMDA).
3. The method of claim 1 wherein the matrix is a polymer selected from the group consisting of polyvinylidene fluoride, polyvinylidene fluoride copolymer, polycarbonate and epoxy.
4. The method of claim 1 wherein the matrix is a highly elastic polymer selected from the group consisting of polyurethane and polysiloxane.
5. The method of claim 1 wherein the matrix is a ceramic selected from the group consisting of silicon dioxides and aluminum oxides.

6. The method of claim 1 wherein the concentration of boron nitride nanotubes in the boron nitride nanotube/polyimide mixture is between 0 and 100% by weight.
7. The method of claim 1 further comprising the step of coating the boron nitride nanotube/polyimide nanocomposite film with metal electrodes.
8. The method of claim 4 wherein the metal for the metal electrode is selected from the group consisting of chrome, gold and a mixture thereof.
9. The method of claim 1 further comprising the step of coating the boron nitride nanotube/polyimide film with compliant electrodes.
10. The method of claim 9 wherein the compliant electrodes are selected from the group consisting of carbon nanotubes, carbon nanotube sheeting, carbon nanotube/polymer composites, gold particles, silver particles and a mixture thereof.

11. A method for forming a boron nitride nanotube/polymer nanocomposite film, comprising:

- synthesizing a high temperature piezoelectric polyimide;
- combining a boron nitride nanotube solution with the high temperature piezoelectric polyimide;
- using a polymer as a matrix; and
- synthesizing a boron nitride nanotube/polyimide nanocomposite film as an electroactive layer.

12. The method of claim 11 wherein the polymer is dianhydride, pyromellitic dianhydride.

13. The method of claim 11 wherein the high temperature piezoelectric polyimide is synthesized from a diamine, 2,6-bis(3-aminophenoxy) benzonitrile ((β -CN)APB) and a dianhydride, pyromellitic dianhydride (PMDA).

14. The method of claim 11 wherein the concentration of boron nitride nanotubes in the boron nitride nanotube/polyimide mixture is between 0 and 100% by weight.

15. The method of claim 11 further comprising the step of coating the boron nitride nanotube/polyimide film with metal electrodes.

16. The method of claim 11 wherein the metal is selected from the group consisting of chrome, gold and a mixture thereof.

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FIG. 1A

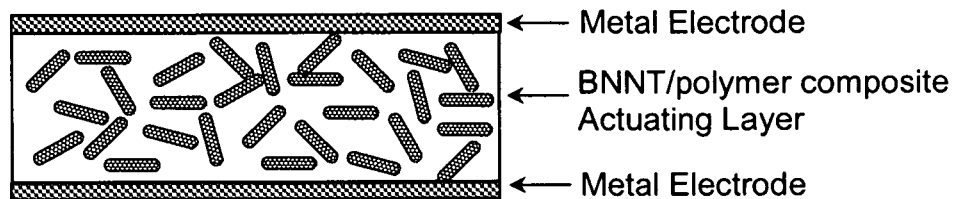
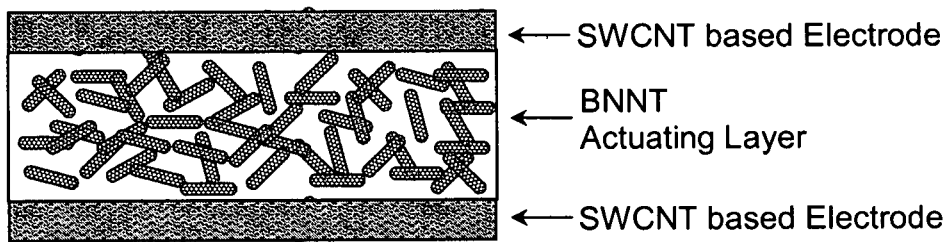


FIG. 1B



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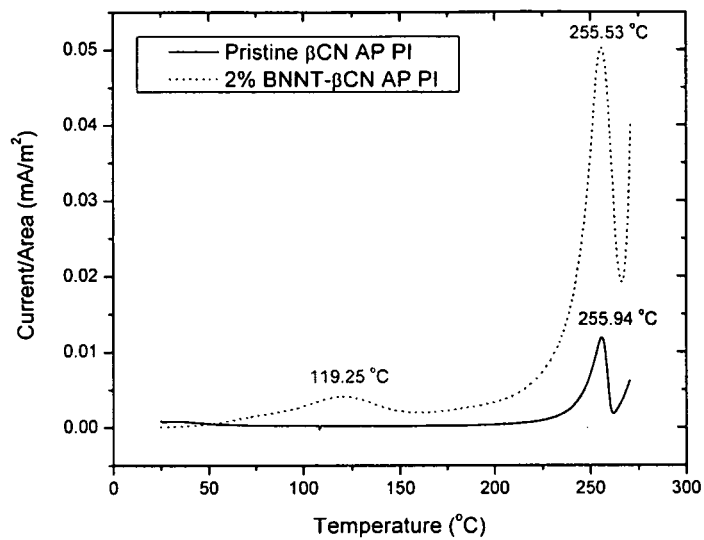


FIG 2A

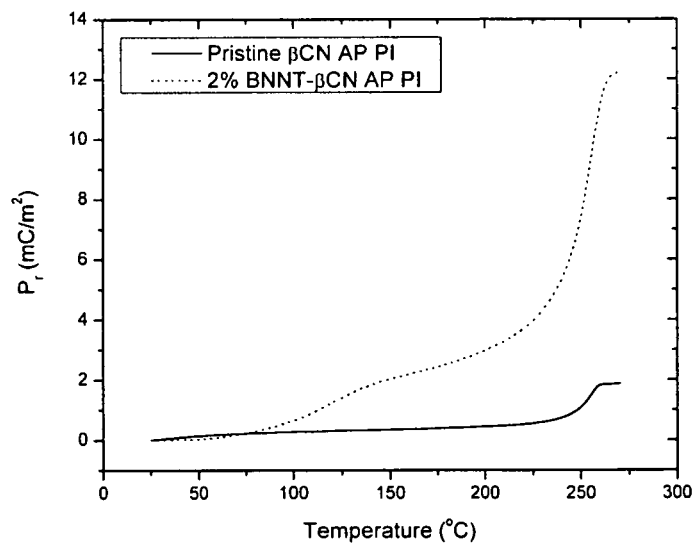


FIG 2B

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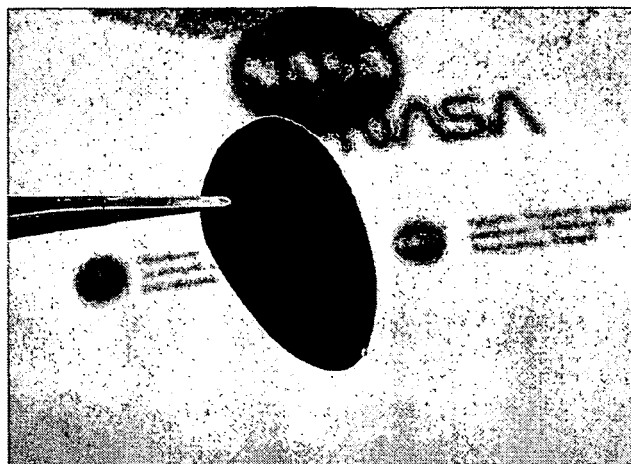


FIG 3

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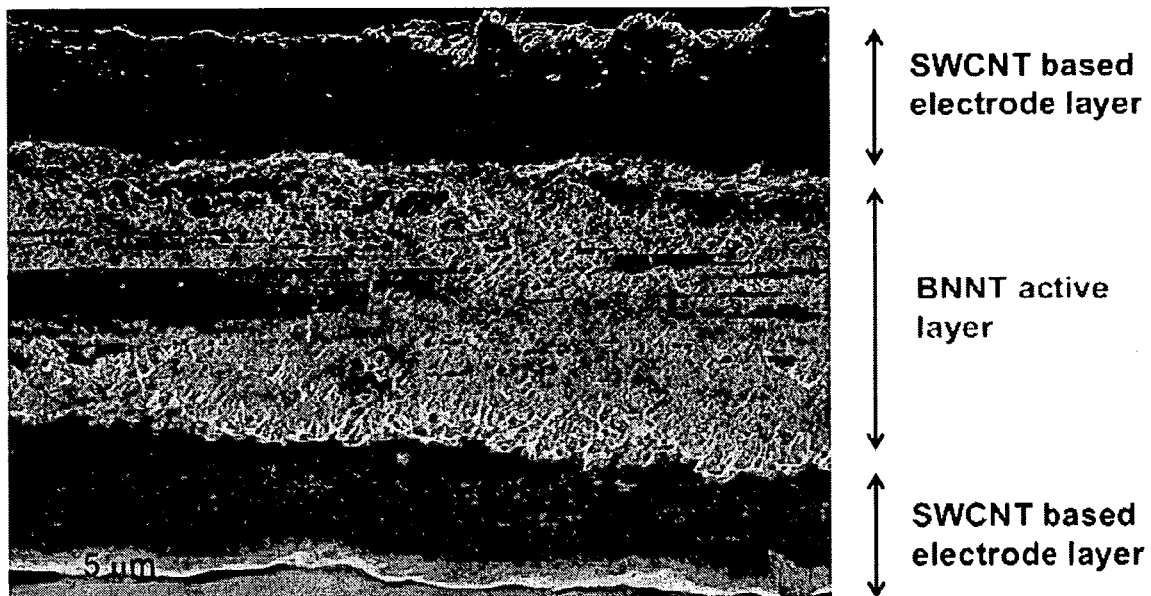


FIG 4

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FIG 5A

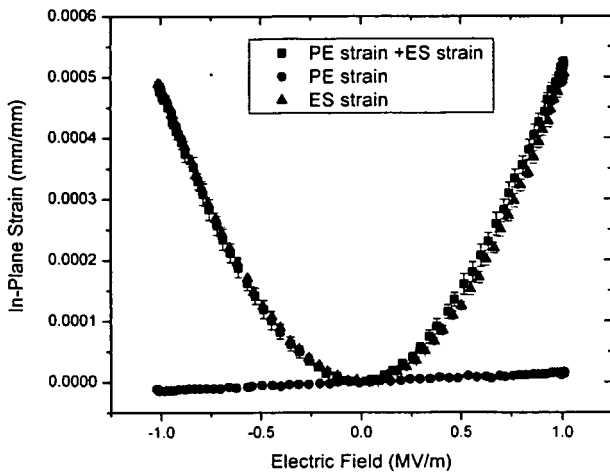


FIG 5B

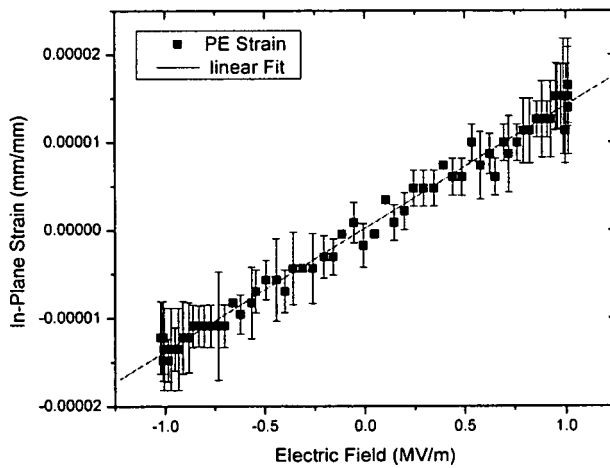
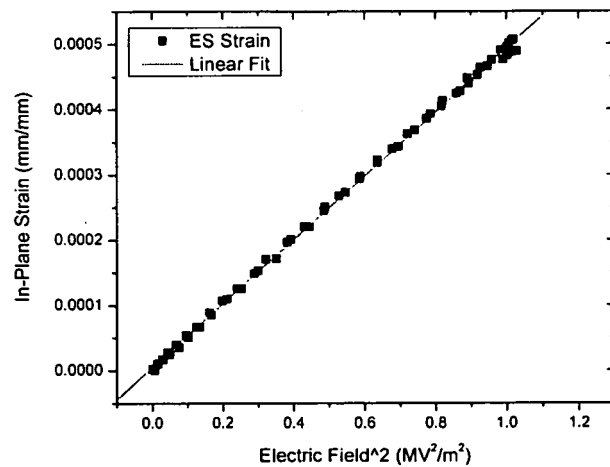


FIG 5C



INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 10/02737

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - H01L 21/00 (2011.01)

USPC - 438/778

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

USPC: 438/778

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

USPC: 438/758, 629, 639, 725, 778 (keyword limited - see search terms below)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

PubWEST (PGPB, USPT, USOC, EPAB, JPAB); GOOGLE; Google Scholar

Terms: bnnt, polymer, nanotube, boron, nitride, electroactive, film, layer, matrix, electrode, concentration.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2009/0221734 A1 (Kuwahara et al.) 03 September 2009 (03.09.2009), entire document, especially abstract, para [0002], [0010], [0023], [0024], [0025], [0041], [0049], [0078], [0080], [0081], [0088], [0116], [0141].	1-16
Y	US 2009/0230353 A1 (Shimazu et al.) 17 September 2009 (17.09.2009), entire document, especially abstract, para [0011], [0012], [0013], [0044], [0050], [0051], [0057].	1-16
P	US 2010/0051879 A1 (Sainsbury et al.) 04 March 2010 (04.03.2010), entire document, especially abstract, para [0003], [0006], [0065], [0067], [0071].	1-16
P	US 2010/0228025 A1 (Cote et al.) 09 September 2010 (09.09.2010), entire document, especially abstract, para [0038], [0040], [0118], [0125], [0177], [0189], [0191], [0193], [0194], [0254], [0301], [0316], [0413].	1-16

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Date of the actual completion of the international search

21 February 2011 (21.02.2011)

Date of mailing of the international search report

07 MAR 2011

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