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(54) **Application of Multilayer Polymer Three-Dimensional Electrode in Energy.**

(57) The invention discloses the application of a multilayer polymer three-dimensional electrode in an energy storage battery. The material is a layered electrode with a single layer of three-dimensional porous structure. Specifically, the three-dimensional porous electrode with multi-layer structure was prepared by scraper coating method and porous material preparation method. The primary purpose of the invention is to prepare an electrode capable of loading an increased amount of active substances by changing the overall structure of the electrode and the components of receptors in the electrode, so as to enhance the cycle stability of the battery electrode, prolong the service life, reduce the production cost and have a broad application prospect.

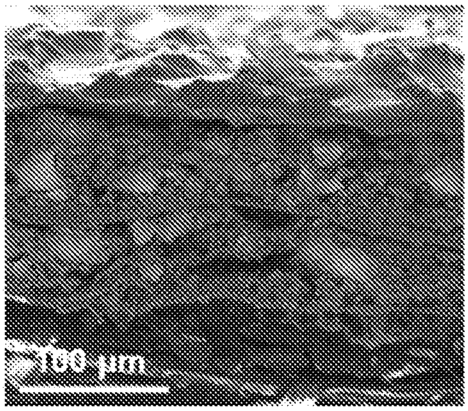

Fig. 1

DESCRIPTION

LU503216

Application of Multilayer Polymer Three-Dimensional Electrode in Energy

FIELD OF THE INVENTION

The invention relates to the fields of material preparation, electrode preparation and electrochemical energy storage, mainly preparing electrodes with layered and single-layer three-dimensional porous structure. Specifically, three-dimensional porous electrodes with multi-layer structure are prepared by scraper coating method and porous material preparation method, and the electrodes are applied to the anode of lithium-sulfur battery and the cathode of lithium-ion battery, so as to improve the electrochemical stability of lithium-sulfur battery and lithium-ion cathode.

BACKGROUND OF THE RELATED ART

In the field of energy storage, battery electrode is one of the most important components in the field of chemical energy storage. However, in the process of electrode reaction, the conversion efficiency of reactants and the total amount of reactants directly affect the energy storage capacity and cycle life of the battery. For example, in a lithium-sulfur battery, the sulfur content of the positive active material directly affects the reversible discharge capacity of the lithium-sulfur battery; the coating amount of spherical carbon anode in various lithium ion batteries also affects the capacity of the whole lithium ion battery. In the preparation process, the amount of active materials in the electrode is usually increased by increasing the coating thickness. However, while increasing the electrode, the impedance of the electrode increases exponentially and the ionic conductivity of the electrode decreases rapidly. These factors seriously restrict the development and commercial application of large-capacity lithium ion batteries.

The invention aims to prepare an electrode capable of loading active substances with increased content by changing the overall structure of the electrode and the components of receptors in the electrode.

SUMMARY OF THE INVENTION

The primary purpose of the invention is to increase the active material loading rate of lithium ion batteries and prepare electrodes with higher loading capacity, so as to overcome the disadvantage of low energy density of lithium ion batteries or lithium sulfur batteries.

The purpose of the invention is realized by the following technical scheme:
The application of polymer multilayer three-dimensional electrode in energy storage battery is characterized in that the material has a three-dimensional multilayer structure, and ternary positive active material and active sulfur material can be loaded inside.
The application of multilayer polymer three-dimensional electrode in energy storage battery is characterized by comprising the following steps and technological conditions:
Step 1: Preparation of three-dimensional carbon materials
(1) Weigh West Bromide and a certain amount of acidic aqueous solution, respectively, according to 1 gram of West Bromide corresponding to 0.5 mol of acidic solution, and dissolve West Bromide in acidic aqueous solution at the reaction temperature of 0-5 degrees Celsius;
(2) After the mixed solution is stirred evenly, the weighed ammonium-containing compound is added according to 1g of ammonium bromide corresponding to 5g of ammonium-containing compound, and a certain amount of micromolecular monomer is measured according to 1g of ammonium-containing compound corresponding to 1-5ml of micromolecular monomer. After stirring for 0.2-2 hours, the measured micromolecule monomer is added, and the obtained solid substance is washed for 3 times with a solution of alcohol and water in any proportion, and finally fully dried in a drying box;
(3) The dried material is heat-treated at a high temperature of 500-900 degrees Celsius for 1-6 hours to obtain the three-dimensional carbon material.
Step 2: Preparation of three-dimensional multilayer electrode and its application in battery.
(1) After the prepared three-dimensional carbon material, active substance and adhesive are weighed according to the mass ratio of (2-4): (3-8): 1, dissolve them in 0.5-2ml solvent with 1ml of adhesive, stir and dissolve, and then add the weighed three-dimensional carbon material and active substance.
(2) Coating the above-mentioned paste-mixed material on aluminum foil with a thickness of 50-200 microns; after drying, uniformly coating a layer of conductive polymer film on the surface of the dried material; repeatedly cross-coating the paste-mixed material and the polymer film; and completely drying to obtain the three-dimensional multilayer electrode.
(3) With the prepared three-dimensional multilayer electrode as anode, metal lithium sheet or sodium sheet as cathode, common lithium ion battery electrolyte as electrolyte and Celgard 2400 as diaphragm, button lithium ion battery or soft package lithium ion battery was prepared.
Compared with the prior art, the invention has the following obvious advantages:

(1) The three-dimensional multilayer electrode prepared by the invention can obviously increase the active material load of the battery, reduce the interface impedance of the battery, improve the dynamic performance of the battery and increase the energy density of the whole battery;

(2) The preparation process of three-dimensional multilayer electrode is optimized, which provides industrial technical support for preparing batteries with higher energy density and better cycle stability.

(3) The invention solves the technical bottleneck of low energy density of lithium ion batteries, and provides technical support for designing practical lithium ion batteries with high energy density.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a cross-sectional scanning electron microscope curve of multilayer polymer three-dimensional electrode prepared in Example 2.

Fig. 2 is the scanning electron microscope curve of the multilayer polymer three-dimensional electrode prepared in Example 3.

Fig. 3 is the scanning electron microscope curve of the three-dimensional electrode of the multilayer polymer prepared in Example 1 after circulation.

Fig. 4 is a scanning electron microscope curve of the multilayer polymer three-dimensional electrode prepared in Example 2.

Fig. 5 is a scanning electron microscope curve of the multilayer polymer three-dimensional electrode prepared in Example 3.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

In order to further explain the technical measures and effects of the present invention, the technical scheme of the present invention will be further explained below with reference to the preferred embodiment of the present invention and its drawings, but the present invention is not limited to the scope of the embodiments.

Example 1

Step 1: Preparation of three-dimensional carbon materials

(1) Weigh West Bromide and a certain amount of acidic aqueous solution, respectively, according to 1g of West Bromide corresponding to 0.5mol of hydrochloric acid solution, and dissolve West Bromide in hydrochloric acid solution in an ice bath environment;

(2) After the mixed solution is stirred evenly, the weighed ammonium sulfate is added according to 1g of ammonium bromide corresponding to 5g of ammonium sulfate, and a certain mass of pyrrole micromolecular monomer is measured according to 1g of ammonium sulfate corresponding to 2ml of pyrrole micromolecular monomer. After stirring for 1 hour, the measured pyrrole micromolecule monomer is added, and the obtained solid substance is washed for 3 times with a solution mixed with alcohol and water in any proportion, and finally fully dried in a drying box;

(3) The dried material was heat-treated at a high temperature of 650℃ for 5 hours to obtain a three-dimensional carbon material.

Step 2: Preparation of three-dimensional multilayer electrode and its application in battery.

(1) After the prepared three-dimensional carbon material, elemental sulfur and binder are weighed according to the mass ratio of 4: 5: 1, 1 ml of binder is dissolved in 2 ml of N-methylpyrrolidone solvent, and the weighed three-dimensional carbon material and elemental sulfur are added after stirring and dissolving.

(2) Coating the paste-mixed material with a thickness of 200 microns on an aluminum foil, drying, uniformly coating a layer of conductive polymer film on the surface of the dried material, repeatedly cross-coating the paste-mixed material and the polymer film, and completely drying to obtain a three-dimensional multilayer electrode.

(3) The button type 2016 lithium-sulfur battery was prepared by using the three-dimensional multilayer electrode prepared above as the positive electrode, metal lithium sheet or sodium sheet as the negative electrode, common lithium-ion battery electrolyte as the electrolyte, and Celgard 2400 as the diaphragm.

Example 2

Step 1: Preparation of three-dimensional carbon materials

(1) Weigh West Bromide and a certain amount of sulfuric acid aqueous solution, respectively, according to 1 gram of West Bromide corresponding to 0.5 mol of sulfuric acid aqueous solution, and dissolve West Bromide in sulfuric acid aqueous solution at the reaction temperature of 0-5 degrees Celsius;

(2) After the mixed solution is stirred evenly, the weighed ammonium persulfate is added according to 1g of ammonium bromide corresponding to 5g of ammonium persulfate, and a certain amount of thiophene monomer is measured according to 1g of ammonium persulfate corresponding to 1-5ml of thiophene monomer. After stirring for 0.3h, the measured

thiophene micromolecule monomer is added, the obtained solid substance is washed with deionized water for 3 times, and finally fully dried in a drying box;

(3) The dried material was heat-treated at a high temperature of 800℃ for 2 hours to obtain a three-dimensional carbon material.

Step 2: Preparation of three-dimensional multilayer electrode and its application in battery.

(1) After the prepared three-dimensional carbon material, lithium manganate and binder are weighed according to the mass ratio of 2: 7: 1, dissolve 1 ml of binder into 0.5 ml of solvent, stir and dissolve, and then add the weighed three-dimensional carbon material and active substance.

(2) Coating the paste-mixed material with a thickness of 100 microns on an aluminum foil, drying, uniformly coating a layer of polythiophene conductive polymer film on the surface of the dried material, repeatedly cross-coating the paste-mixed material and the polymer film, and completely drying to obtain a three-dimensional multilayer electrode.

(3) With the three-dimensional multilayer electrode prepared as the positive electrode, metal lithium sheet or sodium sheet as the negative electrode, common lithium ion battery electrolyte as the electrolyte, and Celgard 2400 as the diaphragm, a soft package lithium ion battery was prepared.

Example 3

Step 1: Preparation of three-dimensional carbon materials

(1) Weigh West Bromide and a certain amount of nitric acid aqueous solution, respectively, according to 1g of West Bromide corresponding to 0.5mol of nitric acid aqueous solution, and dissolve West Bromide in nitric acid aqueous solution at the reaction temperature of 0 degrees Celsius;

(2) After the mixed solution is stirred evenly, the weighed ammonium chloride compound is added according to 1g of ammonium bromide corresponding to 5g of ammonium chloride, and a certain mass of pyrrole micromolecular monomer is measured according to 1g of ammonium chloride compound corresponding to 3ml of pyrrole micromolecular monomer. After stirring for 1.5h, the measured pyrrole micromolecular monomer is added, and the obtained solid substance is washed with alcohol solution for 3 times, and finally fully dried in a drying box;

(3) The dried material was heat-treated at a high temperature of 500℃ for 6 hours to obtain the three-dimensional carbon material.

Step 2: Preparation of three-dimensional multilayer electrode and its application in battery.

(1) After the prepared three-dimensional carbon material, Ferrous lithium phosphate and binder are weighed according to the mass ratio of 2: 8: 1, 1 ml of binder is dissolved in 0.7 ml of solvent, and the weighed three-dimensional carbon material and active substances are added after stirring and dissolving.

(2) Coating the paste-mixed material with a thickness of 150 microns on an aluminum foil, drying, uniformly coating a layer of polypyrrole conductive polymer film on the surface of the dried material, repeatedly cross-coating the paste-mixed and polymer films, and completely drying to obtain a three-dimensional multilayer electrode.

(3) The button lithium-ion battery was prepared by using the three-dimensional multilayer electrode as anode, metal lithium sheet as cathode, common lithium-ion battery electrolyte as electrolyte and Celgard 2400 as diaphragm.

Instruments used for material characterization and electrochemical performance test in the above examples:

Morphology test: Field emission scanning electron microscope and high resolution transmission electron microscope are used, and their equipment names are FEI (scanning electron microscope) and G2 F20FEI Tecnai (high resolution transmission electron microscope).

Charge and discharge test: Wuhan Blue Battery Test System is used, and the maximum range of current and voltage is 10 mA and 5 V respectively.

CLAIMS

1. The application of multi-layer polymer three-dimensional electrode in energy storage battery is characterized in that the material is a layered electrode with a single layer of three-dimensional porous structure, and the main component is a conductive polymer coated layered active material electrode layer. LU503216

2. The application of multilayer polymer three-dimensional electrode in energy storage battery is characterized by comprising the following steps and technological conditions:

Step 1: Preparation of three-dimensional carbon materials

(1) Weigh West Bromide and a certain amount of acidic aqueous solution, respectively, according to 1 gram of West Bromide corresponding to 0.5 mol of acidic solution, and dissolve West Bromide in acidic aqueous solution at the reaction temperature of 0-5 degrees Celsius;

(2) After the mixed solution is stirred evenly, the weighed ammonium-containing compound is added according to 1g of ammonium bromide corresponding to 5g of ammonium-containing compound, and a certain amount of micromolecular monomer is measured according to 1g of ammonium-containing compound corresponding to 1-5ml of micromolecular monomer. After stirring for 0.2-2h, the measured micromolecular monomer is added, and the obtained solid substance is washed with a solution mixed with alcohol and water in any proportion for 3 times, and finally fully dried in a drying box;

(3) The dried material is heat-treated at a high temperature of 500-900 degrees Celsius for 1-6 hours to obtain the three-dimensional carbon material.

Step 2: Preparation of three-dimensional multilayer electrode and its application in battery.

(1) After the prepared three-dimensional carbon material, active substance and adhesive are weighed according to the mass ratio of (2-4): (3-8): 1, dissolve them in 0.5-2ml solvent with 1ml of adhesive, stir and dissolve, and then add the weighed three-dimensional carbon material and active substance.

(2) Coating the above-mentioned paste-mixed material on aluminum foil with a thickness of 50-200 microns; after drying, uniformly coating a layer of conductive polymer film on the surface of the dried material; repeatedly cross-coating the paste-mixed material and the polymer film; and completely drying to obtain the three-dimensional multilayer electrode.

(3) With the prepared three-dimensional multilayer electrode as anode, metal lithium sheet or sodium sheet as cathode, common lithium ion battery electrolyte as electrolyte and

Celgard 2400 as diaphragm, button lithium ion battery or soft package lithium ion battery was prepared.

3. According to the preparation method of multilayer polymer three-dimensional electrode according to Claim 2, in the first step, "West Bromide and a certain amount of acidic aqueous solution are respectively weighed, and 1 gram of West Bromide corresponds to 0.5 mol of acidic solution, and then the West Bromide is dissolved in the acidic aqueous solution at the reaction temperature of 0-5 degrees Celsius", and 1 gram of West Bromide corresponds to 0.5 mol of acidic solution.

4. According to Claim 3, "Dissolve western ammonium bromide in acidic aqueous solution at 0-5 degrees Celsius", the reaction temperature is 0-5 degrees Celsius according to 1 gram of western ammonium bromide corresponding to 0.5 moles of acidic solution.

5. According to Claim 4, "Dissolve West Bromide in Acidic Aqueous Solution", the acid salt solution is preferably hydrochloric acid and sulfuric acid.

6. According to the preparation method of multilayer polymer three-dimensional electrode of claim 2, after the above-mentioned mixed solution is stirred evenly in step 1, according to 1g of western ammonium bromide corresponding to 5g of ammonium-containing compound, the weighed ammonium-containing compound is added. Measure a certain amount of micromolecular monomer according to 1-5 ml of micromolecular monomer corresponding to 1 g of ammonium-containing compound, add the measured micromolecular monomer after stirring for 0.2-2 hours, and clean the obtained solid substance with a solution mixed with alcohol and water in any proportion for 3 times, and finally fully dry it in a drying box. According to "1 g of western ammonium bromide corresponds to 5 g of ammonium-containing compound".

7. According to claim 6, after the above mixed solution is stirred evenly, the ammonium-containing compound in the weighed ammonium-containing compound is preferably ammonium persulfate according to the formula that 1g of western ammonium bromide corresponds to 5g of ammonium-containing compound.

8. According to Claim 6, "Measure a certain amount of micromolecular monomer according to 1 g of ammonium-containing compound corresponding to 1 ~ 5 ml of micromolecular monomer, add the measured micromolecular monomer after stirring for 0.2 ~ 2 hours, and wash the obtained solid substance with a solution of alcohol and water in any proportion for 3 times, Finally fully drying in a drying oven ",the volume of 1 gram of ammonium-containing compound corresponding to micromolecular monomer is 1-5 ml.

9. According to Claim 8, the micromolecular monomers of a certain quality are measured according to 1 g of ammonium-containing compound corresponding to 1-5 ml of micromolecular monomers, and after stirring for 0.2-2 hours, the measured micromolecular monomers are added, and the obtained solid substances are washed for 3 times by a solution mixed with alcohol and water in any proportion, and finally fully dried in a drying box, and the micromolecular monomers are preferably pyrrole and thiophene.

10. According to claim 9, "After stirring for 0.2-2 hours, add the measured micromolecule monomer, and the obtained solid substance is washed for 3 times with a solution of alcohol and water in any proportion, and finally fully dried in a drying box", and the stirring time is 0.2-2 hours.

11. According to the preparation method of the multilayer polymer three-dimensional electrode according to claim 2, the high-temperature heat treatment temperature in the step 1 of "heat treating the dried material at a high temperature of 500-900 degrees Celsius for 1-6 hours to obtain the three-dimensional carbon material" is 500-900 degrees Celsius and the heat treatment time is 1-6 hours.

Revendications

1. D une électrode tridimensionnelle polymère multicouche dans une batterie de stockage d énergie, qui se caractérise par le fait que le matériau est une électrode en couches et que la couche unique est une électrode avec une structure poreuse tridimensionnelle et est principalement composée d un polymère conducteur recouvrant une couche d électrode de matériau actif en couches

2. D une électrode tridimensionnelle polymère multicouche dans une batterie de stockage d énergie, qui se caractérise par le fait que le modèle d utilité comprend les étapes suivantes et les conditions de processus de celle-ci:

Étape 1 : Préparation de matériaux carbone tridimensionnels

(1). Peser respectivement le bromure d'ammonium occidental et une certaine quantité de solution aqueuse acide, en prélevant selon la quantité de 1 gramme de bromure d'ammonium occidental correspondant à 0,5 mole de solution acide, et dissoudre le bromure d'ammonium occidental dans la solution aqueuse acide à la température de réaction de 0~5 degrés Celsius ;

(2). Après agitation uniforme de la solution mélangée, ajouter de composés pesés contenant de l ammonium selon 1 gramme de bromure d'ammonium occidental correspondant à 5 grammes de composés contenant de l ammonium, Selon 1 gramme de composé contenant de l ammonium correspondant à 1~5 ml de petit monomère moléculaire, en ajoutant le petit monomère moléculaire après agitation pendant 0,2~2 heures, nettoyage de la substance solide obtenue avec une solution mélangée avec de l alcool et de l eau dans n importe quelle proportion pendant 3 fois, et enfin séchager complet dans une boîte de séchage ;

(3). Les matériaux carbone tridimensionnels sont obtenus par traitement thermique à 500~900 degrés Celsius pendant 1~6 heures.

Étape 2 : Préparation d électrode multicouche tridimensionnelle et son application dans la batterie

(1). Les matériaux carbone tridimensionnels préparés, les matériaux actifs et les liants sont (2~4) en rapport de masse : (3～8): Après pesée, dissoudre 1 ml de liant dans 0,5~2 ml de solvant, remuer et dissoudre, et ajouter les matériaux carbone tridimensionnels pesés et les substances actives.

(2). Le matériau modifié est revêtu sur la feuille d’aluminium d’une épaisseur de 50~200 microns et, après séchage, un film polymère conducteur est uniformément revêtu à la surface du matériau séché, et le matériau modifié et le film polymère sont recouverts à plusieurs reprises et, après avoir été complètement séchés, une électrode multicouche tridimensionnelle est obtenue.

(3). Une batterie lithium-ion de type bouton ou une batterie lithium-ion souple est préparée en utilisant l électrode multicouche tridimensionnelle préparée comme électrode positive, une feuille de lithium métallique ou une feuille de sodium comme électrode négative, un électrolyte de batterie lithium-ion commun comme électrolyte et un Celgard 2400 comme séparateur.

3. Procédé de préparation d une électrode tridimensionnelle polymère multicouche selon la revendication 2, à l étape 1, «peser respectivement le bromure d'ammonium occidental et une certaine quantité de solution aqueuse acide, en prélevant selon la quantité de 1 gramme de bromure d'ammonium occidental correspondant à 0,5 mole de solution acide, et dissoudre le bromure d'ammonium occidental dans la solution aqueuse acide à la température de réaction de 0~5 degrés Celsius», selon la quantité de 1 gramme de bromure d'ammonium occidental correspondant à 0,5 mole de solution acide.

4. Selon la revendication 3, «dissoudre le bromure d'ammonium occidental dans la solution aqueuse acide à la température de réaction de 0~5 degrés Celsius», selon la quantité de 1 gramme de bromure d'ammonium occidental correspondant à 0,5 mole de solution acide », la température de réaction est de 0~5 degrés Celsius.

5. Selon la revendication 4, «dissoudre le bromure d ammonium occidental dans la solution aqueuse acide», l acide chlorhydrique et l acide sulfurique sont préférés dans la solution aqueuse acide.

6. Procédé de préparation d une électrode tridimensionnelle polymère multicouche selon la revendication 2, à l'étape 1, «après agitation uniforme de la solution mélangée, ajouter de composés pesés contenant de l ammonium selon 1 gramme de bromure d'ammonium occidental correspondant à 5 grammes de composés contenant de l ammonium, Selon 1 gramme de composé contenant de l ammonium correspondant à 1~5 ml de petit monomère moléculaire, en ajoutant le petit monomère moléculaire après agitation pendant 0,2~2 heures, nettoyage de la substance solide obtenue avec une solution mélangée avec de l alcool et de l eau dans n importe quelle proportion pendant 3 fois, et enfin séchager complet dans une boîte de séchage», selon la quantité de 1 gramme de bromure d'ammonium occidental correspond à 5 grammes de composés contenant de l ammonium.

7. Selon la revendication 6, « après agitation uniforme de la solution mélangée, 1 gramme de bromure correspond à 5 grammes de composés contenant de l ammonium en quantité pesée », le persulfate d ammonium est préféré dans les composés contenant de l ammonium

8. Selon la revendication 6, «selon 1 gramme de composé contenant de l ammonium correspondant à 1~5 ml de petit monomère moléculaire, en ajoutant le petit monomère moléculaire après agitation pendant 0,2~2 heures, nettoyage de la substance solide obtenue avec une solution mélangée avec de l alcool et de l eau dans n importe quelle proportion pendant 3 fois, et enfin séchager complet dans une boîte de séchage», le volume de 1 gramme de composé contenant de l ammonium correspondant au petit monomère moléculaire est de 1~5 ml.

9. Selon la revendication 8, «selon 1 gramme de composé contenant de l ammonium correspondant à 1~5 ml de petit monomère moléculaire, en ajoutant le petit monomère moléculaire après agitation pendant 0,2~2 heures, nettoyage de la substance solide obtenue avec une solution mélangée avec de l alcool et de l eau dans n importe quelle proportion pendant 3 fois, et enfin séchager complet dans une boîte de séchage», le pyrrole et le thiophène sont préférés dans le petit monomère moléculaire.

10. Selon la revendication 9, «en ajoutant le petit monomère moléculaire après agitation pendant 0,2~2 heures, nettoyage de la substance solide obtenue avec une solution mélangée avec de l alcool et de l eau dans n importe quelle proportion pendant 3 fois, et enfin séchager complet dans une boîte de séchage», le temps d agitation est de 0,2~2 heures.

11. Procédé de préparation d une électrode tridimensionnelle polymère multicouche selon la revendication 2, à l'étape 1, « les matériaux carbone tridimensionnels sont obtenus par traitement thermique à 500~900 degrés Celsius pendant 1~6 heures», la température de traitement thermique à haute température est de 500~900 degrés Celsius et le temps de traitement thermique est de 1~6 heures.

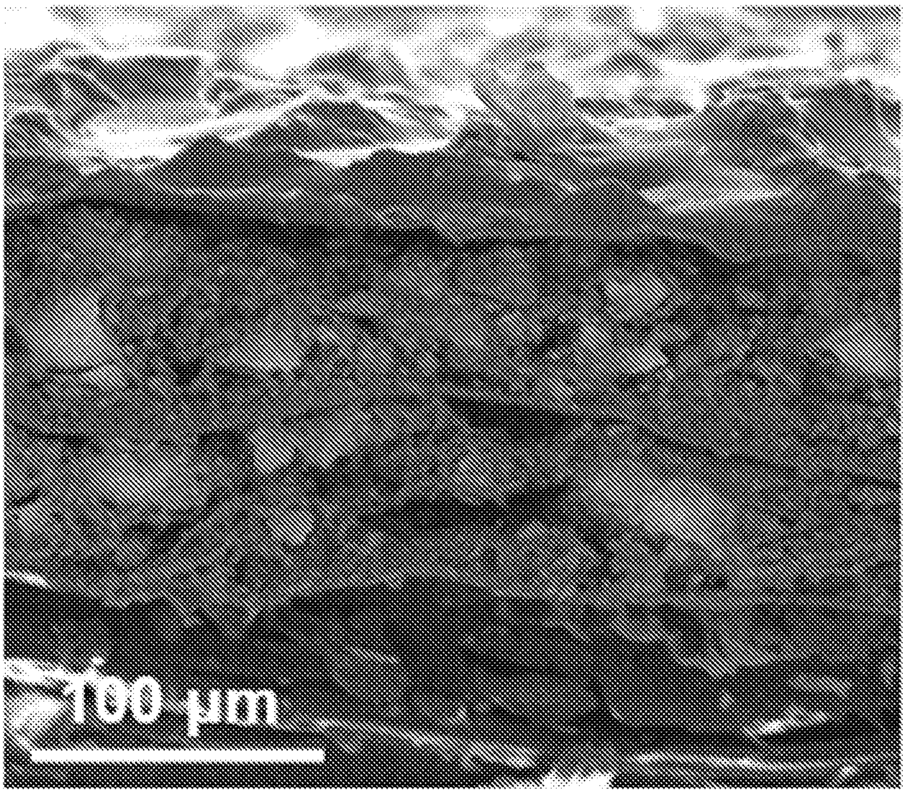

Fig. 1

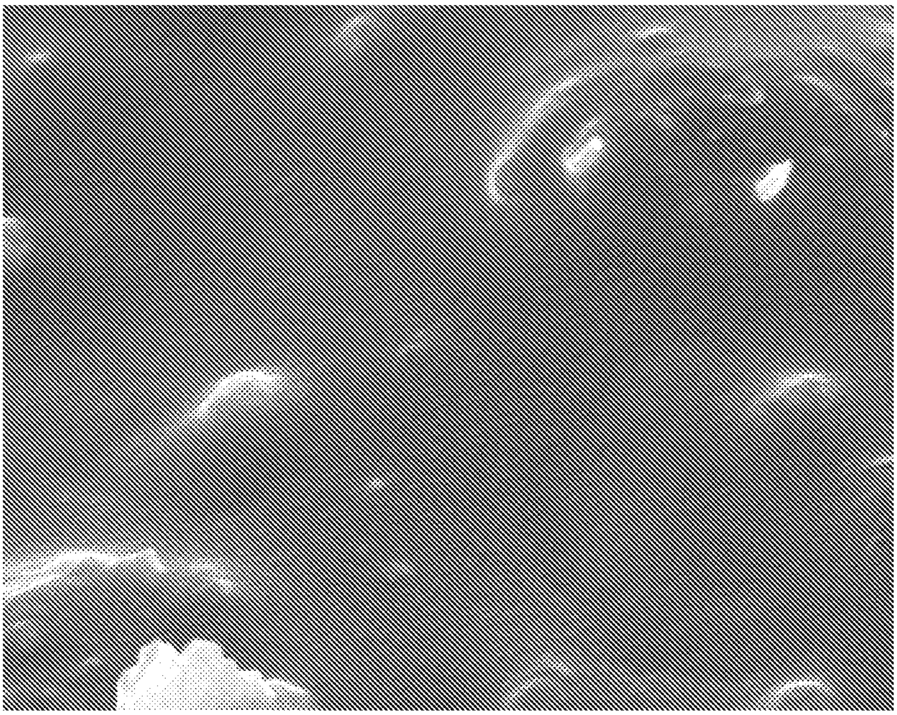

Fig. 2

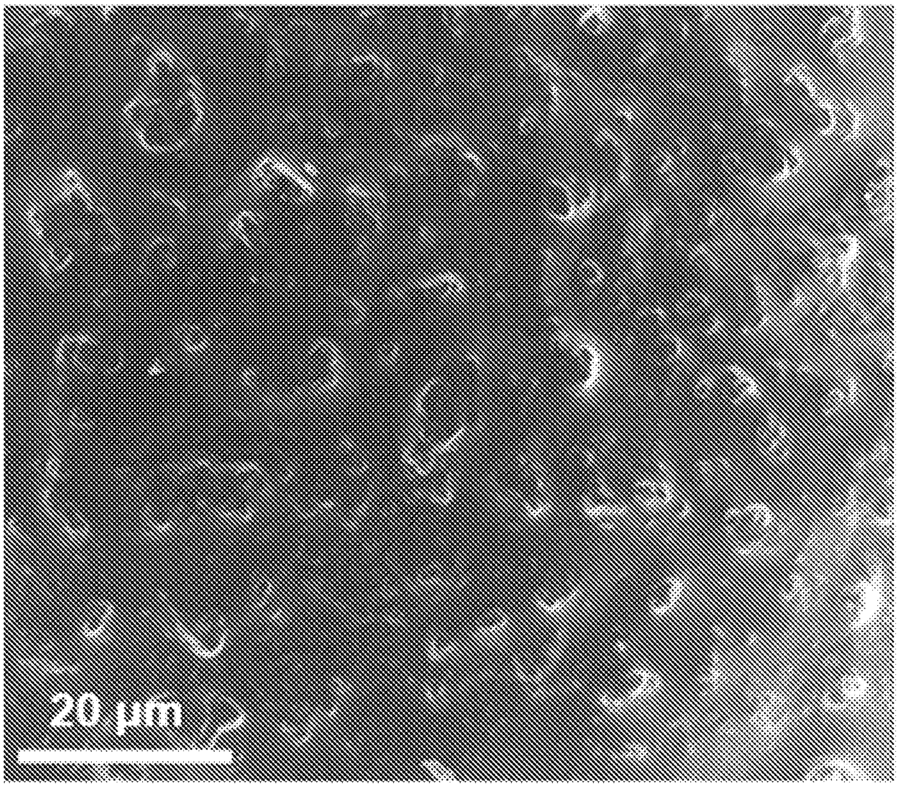

Fig. 3

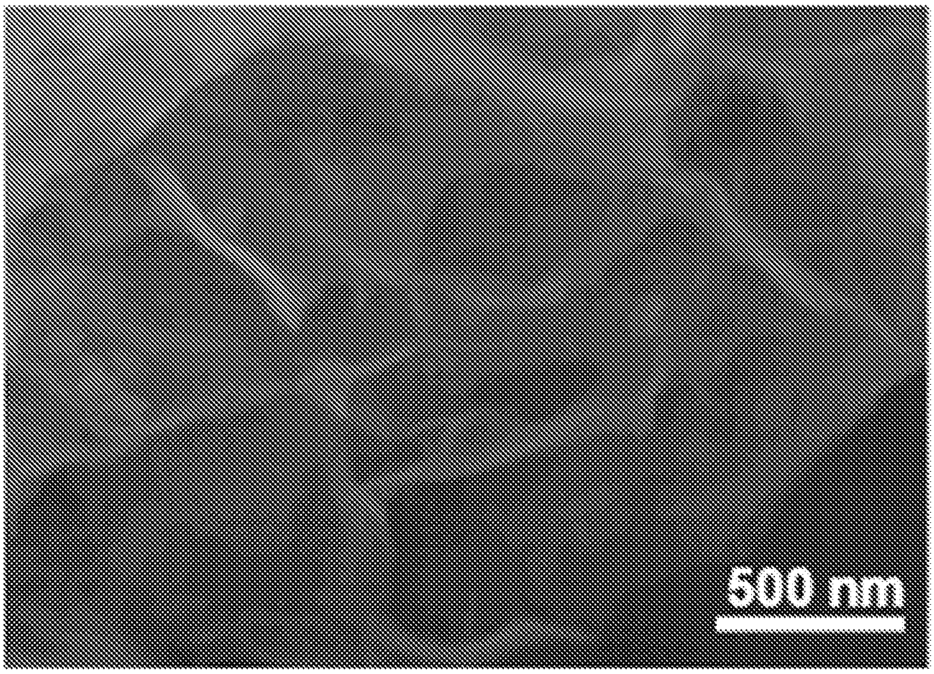

Fig. 4

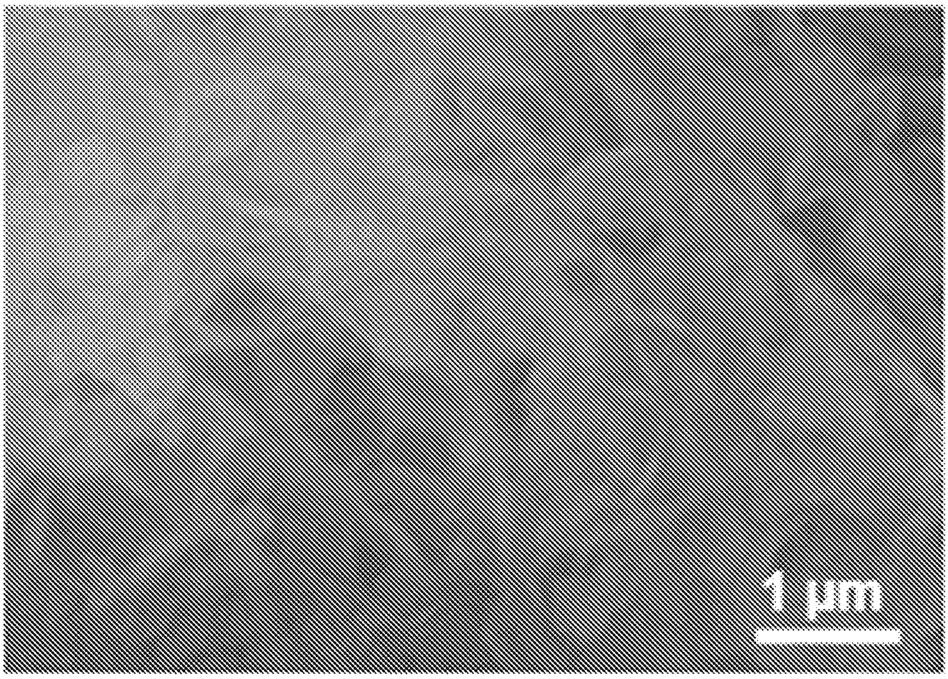

Fig. 5