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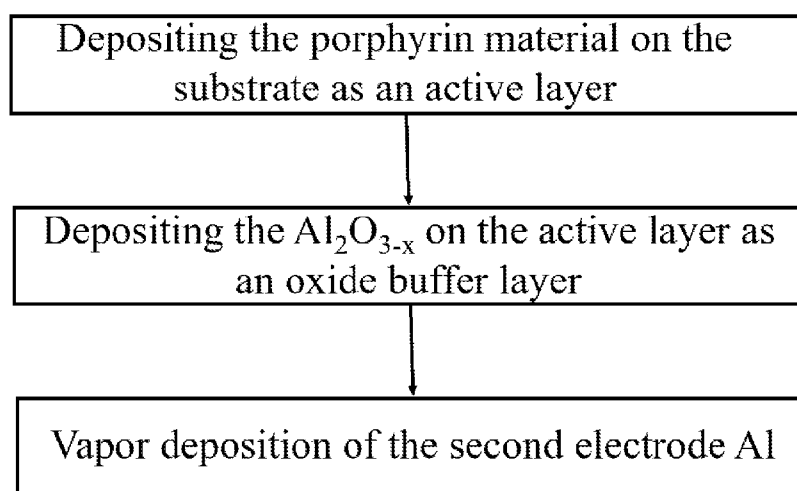


Fig.12

(57) Abstract: A porphyrin memristor device and a method of fabrication of the device are described that include a three-tier structure as anode, switching layer and cathode, the switching layer is interposed between the anode and cathode, also includes porphyrin as active film layer to transfer ions and electrons, switching layer includes an oxide buffer layer to provide oxygen ions. In an implementation, the active film layer comprises molecular materials containing porphyrin basic structural unit, the oxide buffer layer may be aluminum oxide (Al₂O_{3-x}). The porphyrin memristor can be applied to a cross-array artificial neural computing system, and an array is used as a node linking processor to manufacture an artificial intelligence system having learning function.



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A PORPHYRIN MEMRISTOR AND THE FABRICATION METHOD THEREOF

BACKGROUND

Memristor is a new type of electronic components with its unique non-linear electrical properties, independent of resistors, capacitors, inductors and other basic components, is widely regarded as the fourth basic passive electronic components, its concept was first proposed by the Chinese scientist Professor Cai Shaotang in 1971. In 2008, HP Labs verified it and published its experimental result in "Nature" magazine, and filed US patent US2008 / 0090337A1 which had been granted thereafter. Although memristor has shown advantage in overcoming the von Neumann architecture, low power consumption and parallel processing, boasting wide application potential in the fields of basic circuit design, new memory, logic circuit and artificial intelligence device, however, it is still faced with commercial bottlenecks in such device property areas as tolerance, reliability, and batch reproducibility. At present, the mechanism of memristor still remains to be further explained and investigated systematically, the research activities still mainly focused on inorganic materials, while no breakthrough has been realized with respect to organic materials.

So far, important progress has been made in the field of inorganic memristors, which can improve the performance of the device by controlling the composition of the material, the phase of the mixture, and the design in device and even the circuit level. For an example, tantalum oxide memristor has shown more than 10 billion switching cycles, which can improve the stability of the intermediate state through the mixed phase, thus achieving multilevel storage, feedback mechanism and refresh technology can also effectively improve its tolerance.

Nevertheless, there are still a lot of problems for inorganic memristors in terms of processing mechanisms, to name a few of them, poor repeatability of the device, poor reliability, poor batch consistency, not suitable for flexible production process and high energy consumption etc. Compared with inorganic materials, organic materials has such advantages as being flexible, low-cost processing, large area, easy to be integrated into daily necessities, it is in line with the future trends of mobile Internet, large data, artificial intelligence and cloud computing . Despite all listed above, however, the actual application for organic memristor still doesn't really commence, considering the fact of the slow progress of materials and devices, and lack of organic memristor media design.

The ideal organic memristor is a kind of organic electronic ion device, which can effectively improve the tolerance, stability, maintainability, controllability and repeatability of the device. It can be used by a wide range of flexible electronic circuit devices, such as memory, switches and logic circuits and function units. For instance, the organic film memristor may be used as the closed or open switch of interleaving memory. So far, organic memristors of small molecules, macromolecules and nanomaterials have been reported, but the organic memristors with a definite molecular mechanism has not emerged, moreover, many problems remain to be

further clarified with respect to evaluation and standard.

BRIEF DESCRIPTION OF THE DRAWINGS

The detailed description is described with reference to the accompanying figures, in which like reference numerals may correspond to similar, though perhaps not identical, components. For the sake of brevity, reference numerals having a previously described function may or may not be described in connection with other figures in which they appear.

FIG. 1 is a structural view illustrating an embodiment of porphyrin type memristor device.

FIG.2. illustrates the molecular structure of the active film layer used in the switching layer of porphyrin type memristor.

FIG. 3 illustrates the hysteretic I-V loops of ZnTPP memristor with a moderate thickness of Al₂O₃-x layer measured at 10 V with voltage-sweep rates of 0.1 Vs-1.

FIG. 4 is the typical counter figure-eight hysteretic loop current-voltage (I-V) characteristics of a ZnTPP memristor with the moderate thickness of Al₂O₃-x layer.

FIG.5 illustrates the hysteretic I-V loops of ZnTPP memristor with the moderate thickness of Al₂O₃-x layer measured at 10V after the device being stored in the air for one year.

FIG. 6 illustrates the hysteretic I-V loops of ZnTPP memristor without Al₂O₃-x layer measured at 10 V.

FIG. 7 illustrates the hysteretic I-V loops of the memristor only with Al₂O₃-x layer measured at 10 V.

FIG. 8 illustrates the hysteretic I-V loops of TPP memristor with the moderate thickness of Al₂O₃-x layer measured at 10 V.

FIG. 9 illustrates the hysteretic I-V loops of NiTPP memristor with the moderate thickness of Al₂O₃-x layer measured at 10 V.

FIG. 10 illustrates the hysteretic I-V loops of CoTPP memristor with the moderate thickness of Al₂O₃-x layer measured at 10 V.

FIG. 11 illustrates the hysteretic I-V loops of FeClTPP memristor with the moderate thickness of Al₂O₃-x layer measured at 10 V.

Fig.12 is a flow diagram illustrating an example process of fabricating porphyrin memristor.

DETAILED DESCRIPTION

Reference is now made in detail to specific embodiments of the disclosed porphyrin memristor based on single dual electron/ion transport mechanism and specific examples of ways for creating the disclosed porphyrin memristor based on single dual electron/ion transport mechanism. When applicable, alternative embodiments are also briefly described.

As used herein, the singular forms "a," "an," and "the" include plural referents unless the context clearly dictates otherwise.

As used in this specification and the appended claims, "approximately" and "about" mean a $\pm 10\%$ variance caused by, for example, variations in manufacturing processes.

In the following detailed description, reference is made to the figures accompanying this disclosure, which illustrate specific examples in which this disclosure may be practiced. The components of the examples can be positioned in a number of different orientations and any directional terminology used in relation to the orientation of the components is used for purposes of illustration and is in no way limiting. Directional terminology includes words such as "top," "bottom," "front," "back," "leading," "trailing," etc. Herein, the term 'about' when applied to a value generally means plus or minus 10% unless otherwise expressly specified. Moreover, examples herein are intended to be illustrative only and are presented for discussion purposes and not by way of limitation.

It is to be understood that other examples in which this disclosure may be practiced exist, and structural or logical changes may be made without departing from the scope of the present disclosure. Therefore, the following detailed description is not to be taken in a limiting sense. Instead, the scope of the present disclosure is defined by the appended claims.

FIG. 1 is a structural view illustrating an embodiment of porphyrin type memristor device. A memristor, based on the structure of ITO/MTPP/Al₂O₃-x/Al with electron/ion transport mechanism, forms the first electrode, the switching layer and the second electrode from bottom to top in turn. Wherein the first electrode and the second electrode are electrically connected to an external power source, while the switching layer is used to achieve the conversion between the resistance state. Wherein the first electrode is indium tin oxide, the switching layer includes a porphyrin active layer and an oxide buffer layer, the second electrode is aluminum. The porphyrin memristor further comprises an electrode lead-out layer for drawing out the first electrode and the second electrode, respectively, and is electrically connected to the external power source; and the electrode extraction layer is metal gold. The porphyrin memristor further comprises a substrate layer underlying the first electrode layer, the material of the substrate layer being glass.

The substrate is typically made of silicon dioxide, doped silica or other materials. Wherein the first electrode and the second electrode material are independently selected from indium tin oxide, aluminum, copper, and gold, wherein the first electrode is preferably indium tin oxide. The second electrode has a thickness of about 100 nm. The second electrode may be formed by a physical vapor deposition method such as vapor deposition. The oxide buffer layer is made of a metal oxide or a mixed metal oxide, preferably an aluminum oxide, a titanium oxide, a zirconium oxide, a hafnium oxide, a molybdenum oxide, an oxide of a molybdenum oxide and a mixed metal oxide which is capable of producing an oxygen anion material composition including oxygen ions, Indium gallium zinc oxide. The thickness of the oxide buffer layer is about 5 nm. The oxide buffer layer may be formed by a physical vapor deposition method such as vapor deposition. The active layer is a molecule containing porphyrin basic structural unit, including porphyrin small molecule, porphyrin macromolecule and porphyrin-containing nano-materials.

Fig. 2 is a schematic view showing the structure of the porphyrin material of the porphyrin type memristor active film layer. The porphyrin iron in hemoglobin can transport oxygen through reversible coordination with oxygen, so that the organism provides energy for metabolic production through aerobic respiration. Therefore, porphyrin materials have the potential to transport oxygen ions. Based on this, we screened five porphyrin materials for study.

Fig. 3 shows the hysteretic I-V loops of ZnTPP film measured at 10 V and -10V with voltage-sweep rates of 0.1 Vs-1. Smooth hysteretic I-V curves without abrupt jump were observed during the bidirectional voltage sweeps.

Neural synapses can actually be seen as two-terminal devices, and it has a unique nonlinear transmission characteristic. The strength of the connection between the neurons determines the transfer efficiency, which can be dynamically changed with the training of the stimulus signal or the suppression signal, and the state of the continuous change is maintained. Memristors have a resistance that varies continuously with the amount of electricity flowing through them. This non-linear electrical property has a high degree of similarity to the nonlinear transmission characteristics of synapses. When the bias voltage sweeps from 0 to 10 V, the current increases nonlinearly and rises rapidly at about 7 V, which signifies an apparent transport enhancement of carriers. The ultimate current level increases to ~1100 μ A at 10 V. During the back scanning from 10 V to 0, the anticlockwise hysteresis appears, which can be ascribed to the ionic species motion or migration. During the following negative scanning from 0 to -10 V, a negative differential resistance (NDR) performance can be observed with a peak voltage at about -6.5 V. In this memristor, if we consider the conductivity of the device as a synaptic weight, the above results show a similarity to the nonlinearity of the synaptic transmission in biological neurons. Wherein a synapse is stimulated / suppressed by applying a positive voltage / a negative voltage. The memristor has similar electrical characteristics as the existing memristor.

Fig. 4 illustrates the 100 experimental switching I-V loops of ZnTPP at 10 V and -10 V. The 100 experimental switching loops traversed as figure-of-eights were detected, which shows a high degree of repeatability without any saltation, and reveals that our device can maintain normal working condition during long operating time. After more than 100 cycles, the device curve could not change obviously that suggested that device is reliable strongly. The memristor has similar electrical characteristics as the existing memristor. At the same time, the current-voltage characteristic of the device remains constant for 100 times, which indicated that the stability of the device was good.

Fig. 5 is the hysteretic I-V loops of ZnTPP film measured at 10 V and -10 V after store in ambient atmosphere one year. As expected, the device still shows remarkable performance, albeit current higher than its fresh counterparts. The device after one-year storage also shows stable IV curve that are excellent for the simulation and application. It can be seen that the memory device provided by the invention has good stability, which can reduce the packaging requirement to a certain extent and has the characteristics of good stability.

Fig. 6 is the hysteretic I-V loops of the porphyrin memristor without forming the oxide buffer layer. It shows the typical IV curves without obvious hysteresis loop during 15 V ~ -15 V while the rectification characteristics are remained with a current of 3440 μ A at 15 V. The device with this structure doesn't have the similar electrical characteristics as the existing memristor. It can be seen that the oxide buffer layer in the device structure plays an essential role, which improves the performance of the device significantly by using the oxide Al₂O₃-x as the modifier layer.

Fig. 7 illustrates the hysteretic I-V loops of the device without porphyrin layer to investigate the essential role of the ZnTPP active layer. The device conductive level is still kept at the HRS and doesn't show the typical figure-eight hysteretic loop, which doesn't have the similar electrical characteristics as the existing memristor. It can be seen the essential role of coordination of ZnTPP in modulating the diffusion space of oxygen ions efficiently and controllably to achieve complex neurological multifunctional simulations.

Fig. 8 to Fig. 11 illustrate the hysteretic I-V loops of TPP, NiTPP, CoTPP and FeTPP-Cl. The conductivity of TPP is very strong, and there is a phenomenon that the conductivity increases first and then decreases. This is because there is no bond of bond and oxygen ion migration is too fast. The activity of metallic Co is stronger than that of nickel Ni, and the coordination bond with oxygen ions is more unstable. With the increase in metal lively, the device conductivity increased. Mainly because the metal is more active, and oxygen ions coordination bond more unstable, so the more easily migrate oxygen ions. FeTPP-Cl rough IV curve, and poor conductivity because the Cl occupied the oxygen coordination site, thereby reducing the probability of

oxygen coordination, making it difficult to migrate. FeTPP-Cl rough IV curve, and poor conductivity because the Cl occupied the oxygen coordination site, thereby reducing the probability of oxygen coordination, making it difficult to migrate.

Fig.12 is the preparation method flow chart of porphyrin memristor. The manufacturing method can be used to prepare the memristors of the five embodiments. The specific process of the production method will be described below. The indium tin oxide conductive glass serves as a substrate and a first electrode of the device. Before the preparation, the indium tin oxide conductive glass is sequentially cleaned by acetone, ethanol and deionized water, then ultrasonic cleaned by acetone, ethanol and deionized water in three steps and then dried. The indium tin oxide glass treated in the above step was subjected to UV ozone treatment for 5 minutes. The ITO glass treated in the above was placed in a vacuum evaporation MTPP of about 25 nm, Al₂O₃-x of about 5 nm and Al electrode of about 100 nm, when the cabin pressure is less than 5×10^{-5} Pa; wherein the material of the vapor-deposited active layer is MTPP, the speed of the evaporation is about 0.5 Å/s, the thickness is about 10-15 nm by using crystal-controlled; wherein the material of the vapor-deposited Al₂O₃-x layer is Al, the speed of the evaporation is about 0.1-0.3 Å/s, which is formed by an autoxidation reaction, the thickness is about 5 nm; the speed of the Al-evaporation is about 5 Å/s; the thickness of the film is measured using a step meter and further confirmed by STEM. After completion of the coating, the electrode was cooled to room temperature while maintaining the vacuum state, and then subjected to an electric test.

What is claimed is:

1. A porphyrin memristor, comprising: a three-tier structure as anode, switching layer and cathode, said switching layer, interposed between said anode and cathode, includes porphyrin as active film layer to transfer ions and electrons, switching layer also includes an oxide buffer layer to provide oxygen ions.

2. The porphyrin memristor of claim 1, wherein said anode is the first electrode formed in tin indium oxide, said cathode is the second electrode formed in aluminum, three-tier structure consists of the first electrode, switching layer and the second electrode, from bottom to top, the first electrode and the second electrode connect to external power source, the switching layer serves to the purpose of conversion of multilevel resistance state.

3. The porphyrin memristor of claim 1, wherein the thickness of said oxide buffer layer is in the range of several nanometers to hundreds of nanometers, oxide buffer layer comprises of material which can generate oxygen anion.

4. The porphyrin memristor of claim 3, wherein the material which can generate oxygen anion includes metal oxide containing oxygen ion or mixed metal oxide.

5. The porphyrin memristor of claim 1, wherein the active film layer comprises molecular materials containing porphyrin basic structural unit.

6. The porphyrin memristor of claim 5, wherein the molecular materials include porphyrin small molecules, porphyrin macromolecules and porphyrin-containing nanomaterials, molecules containing porphyrin basic structural unit is porphyrin containing metal coordination and its derivatives.

7. The porphyrin memristor of claim 1, wherein the active film layer may include porphyrin zinc (ZnTPP), porphyrin iron (FeTPP), porphyrin nickel (CoTPP) or porphyrin nickel (NiTPP).

8. The porphyrin memristor of claim 1, wherein the anode includes tin indium oxide and a substrate, with tin indium oxide formed above the substrate, the active film layer over tin indium oxide is made of porphyrin materials, the thickness of the active film layer is between 10-200 nm, the thickness of the oxide buffer layer is between 5-100 nm, the oxide buffer layer is aluminum oxide, on top of which is coated by vaporization with aluminum layer of 100-500 nm to function as cathode.

9. The porphyrin memristor of claim 1, wherein the transmission mechanism of the device belongs to single dual electron/ion transport mechanism; wherein the volt-ampere curve of the device shows the characteristic of "8" shape hysteresis; wherein the device exhibits a voltage dependent Matthew effect.

10. The porphyrin memristor of claim 1, wherein the first electrode and the second electrode each may include a material independently selected from the group consisting of Indium gallium oxide (ITO), aluminum, molybdenum, niobium, copper, gold, palladium, platinum, tantalum, ruthenium, ruthenium oxide, silver, tantalum nitride, titanium nitride, tungsten and tungsten nitride.

11. The porphyrin memristor of claim 3, wherein said material may be aluminum oxide ($\text{Al}_2\text{O}_{3-x}$), titanium oxide, indium gallium zinc oxide (ITO), hafnium oxide, molybdenum oxide or zirconium oxide.

12. A method for fabricating the porphyrin memristor of claim 1, comprising: providing the anode, forming the switching layer on top of the anode, then forming the cathode on top of the switching layer, the metal oxide buffer layer is formed through low vacuum level evaporation method to improve the quality of the switching layer, ensuring that the oxygen content is lower than the integer ratio.

13. The method of claim 12, wherein to form the porphyrin memristor based on the single dual electron/ion transport mechanism, comprising:

Step1: Using the conductive glass (ITO) as the substrate and anode, then rinse them with acetone, ethanol and ultra-clean water consecutively, and followed by a three-step ultrasonic cleaning and drying with acetone, ethanol, and ultra-pure water;

Step 2: subjected the dried ITO glass to UV ozone treatment for 5 minutes;

Step 3: place the ITO glass processed in step 2 in a vacuum evaporation system, adjust the cabin pressure to less than 5×10^{-5} Pa, followed by the evaporation process of MTPP of about 25 nm, $\text{Al}_2\text{O}_3\text{-x}$ of about 5 nm and Al electrode of about 100 nm, respectively, the speed of the MPTT evaporation is about $0.5 \text{ \AA/s}$, the thickness is about 10-15 nm by crystal-controlling; wherein the speed of the evaporation to form $\text{Al}_2\text{O}_3\text{-x}$ is about $0.1\sim 0.3 \text{ \AA/s}$, through autoxidation reaction of AL, the thickness of $\text{Al}_2\text{O}_3\text{-x}$ is about 5 nm; the speed of the Al electrode evaporation is about $5 \text{ \AA/s}$; the thickness of the film is measured by step meter and further confirmed by STEM;

Step 4: when coating process finished, the electrode was cooled to room temperature while maintaining the vacuum state, and then subject the memristor to electric test.

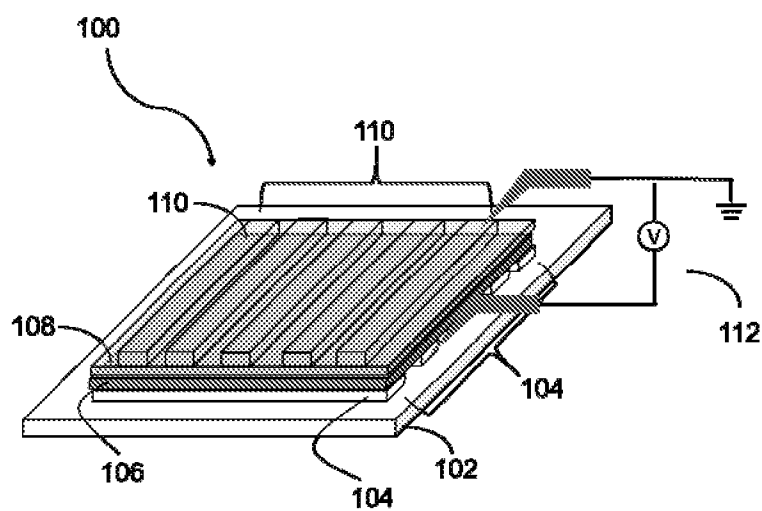
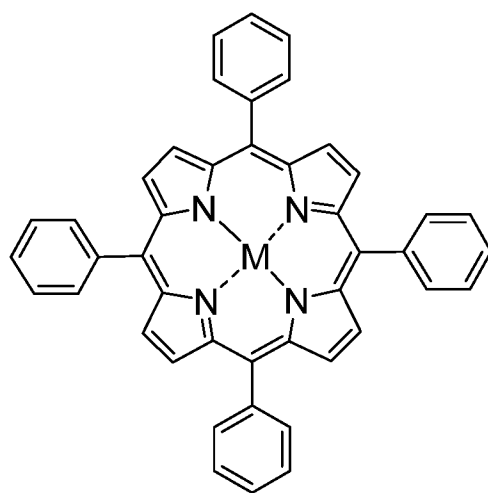


Fig. 1



MTPP

Without M: TPP
 M= Zn: ZnTPP
 M= Ni: NiTPP
 M= Co: CoTPP
 M=Fe-Cl: FeTPPCL

Fig. 2

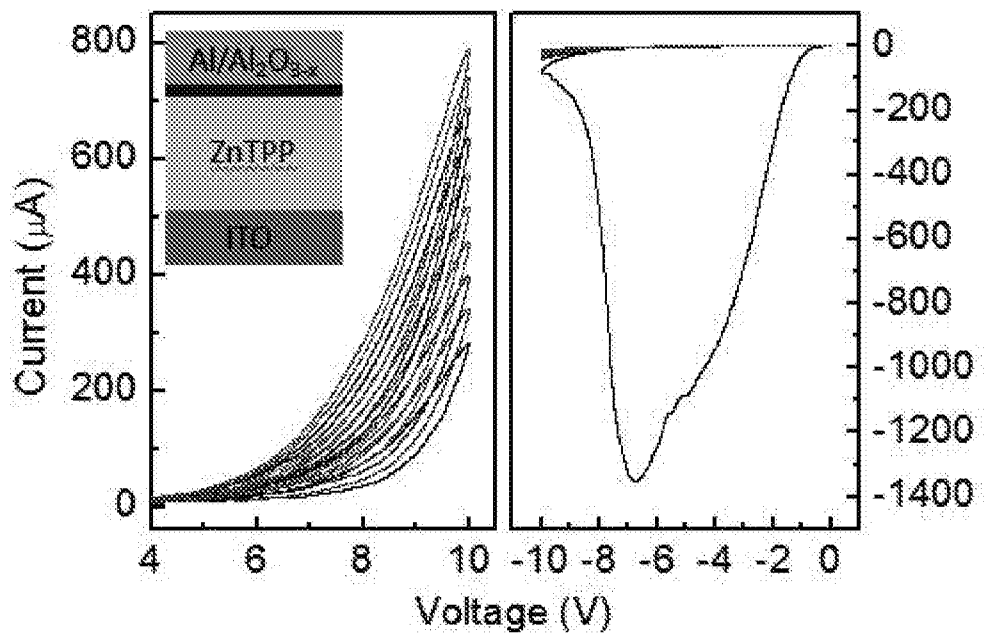


Fig.3

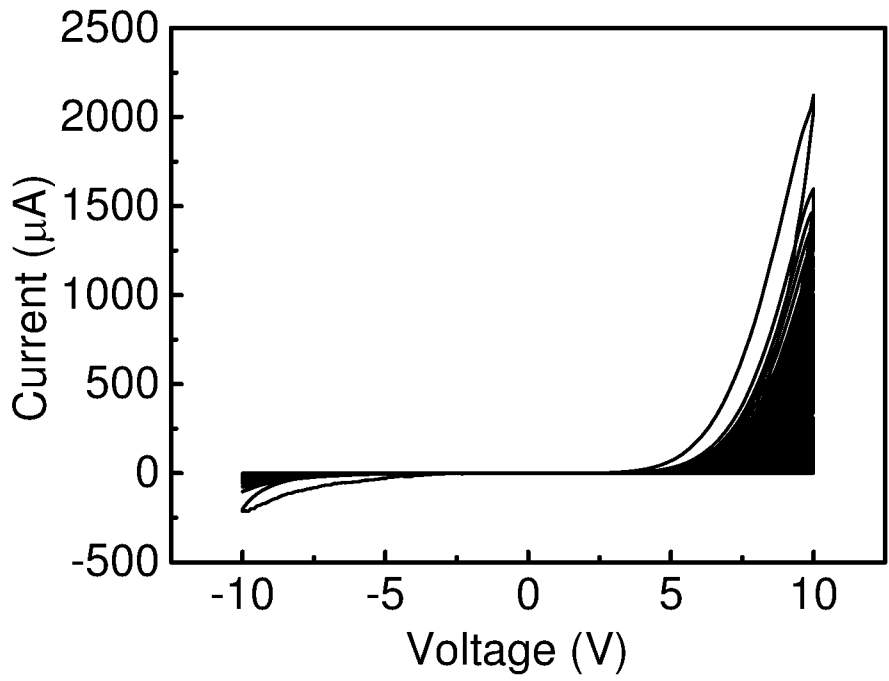


Fig.4

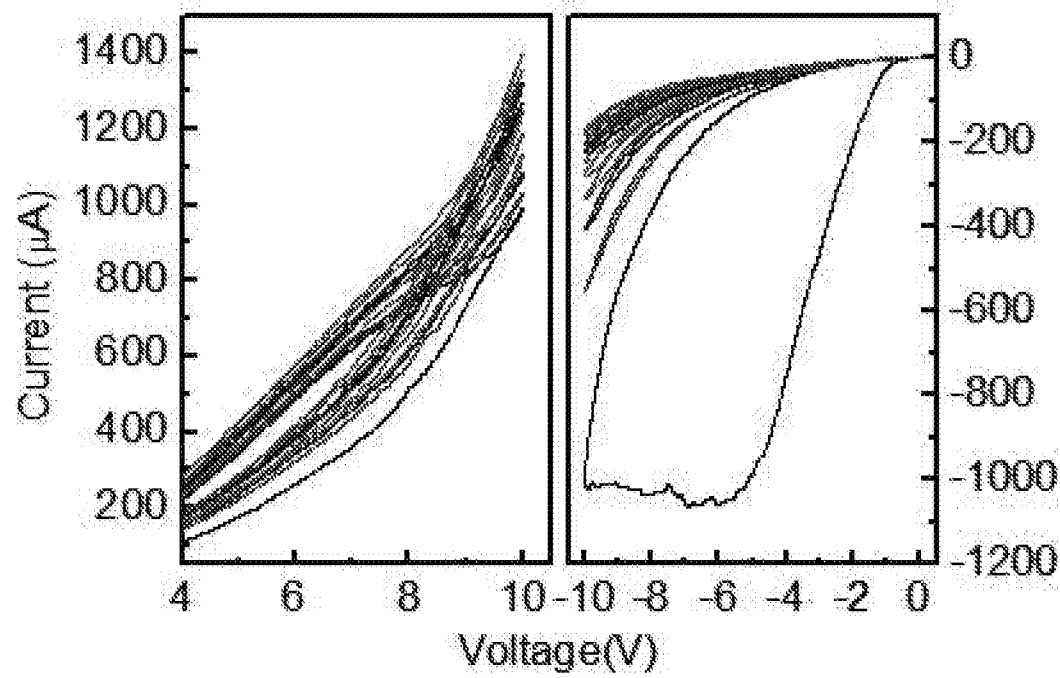


Fig.5

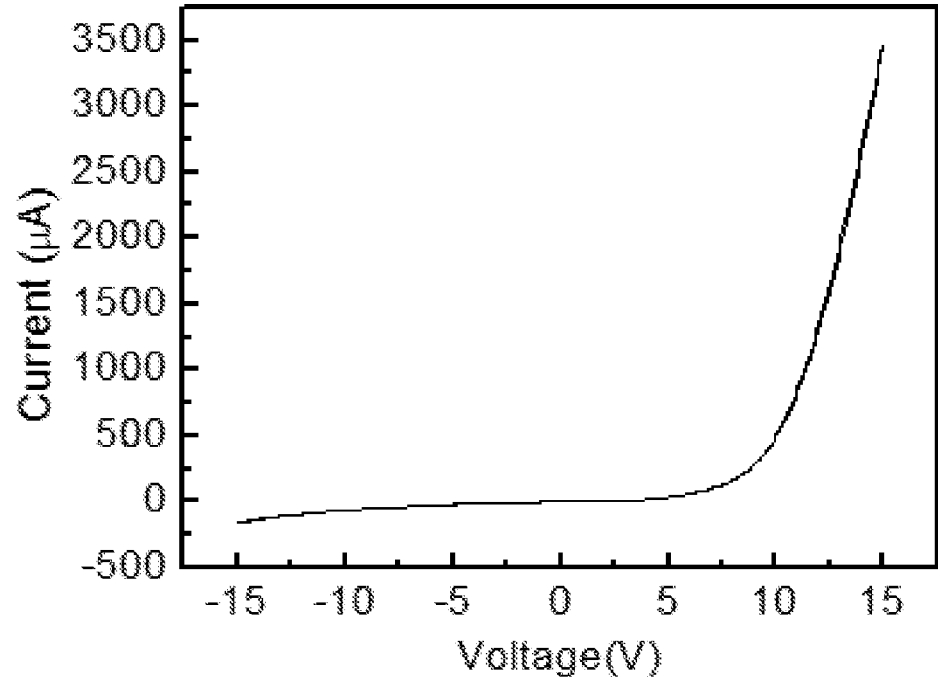


Fig.6

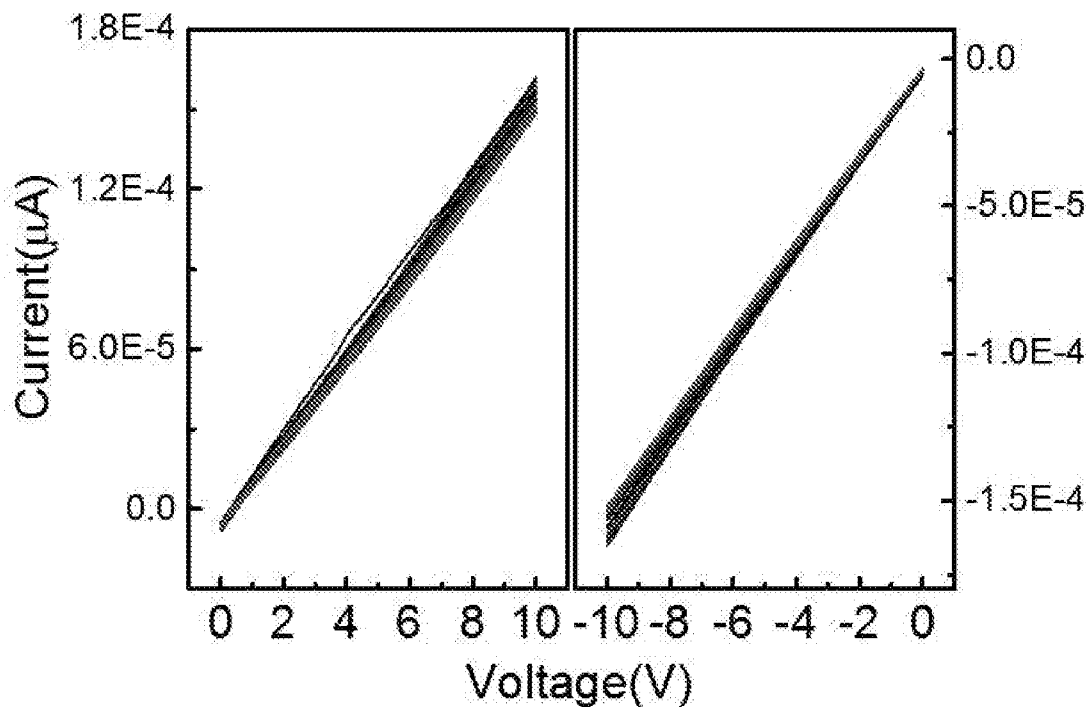


Fig.7

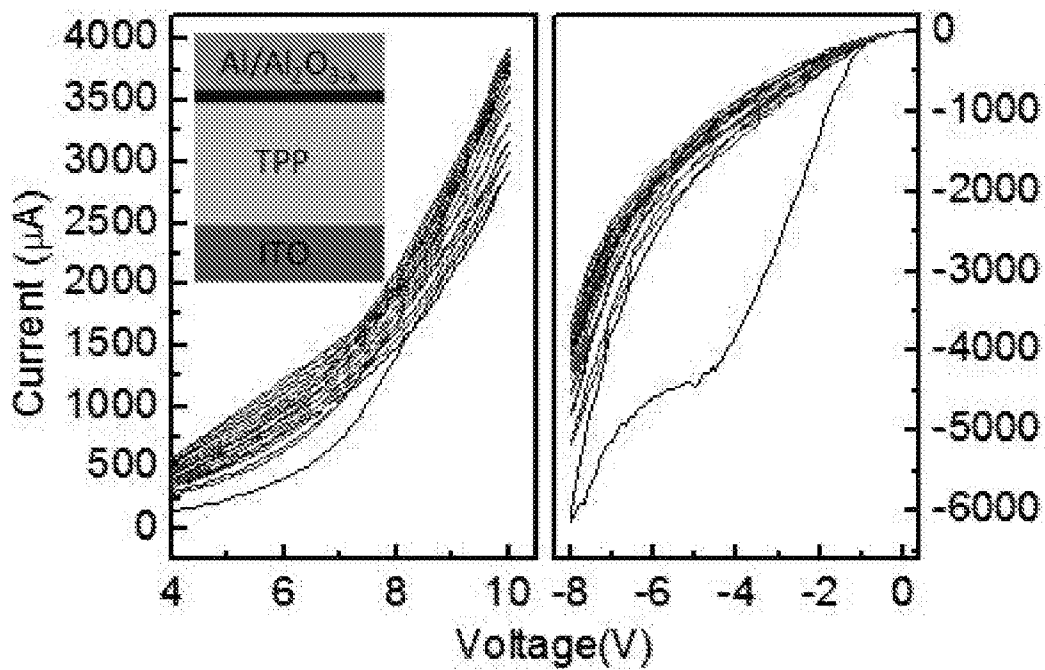


Fig.8

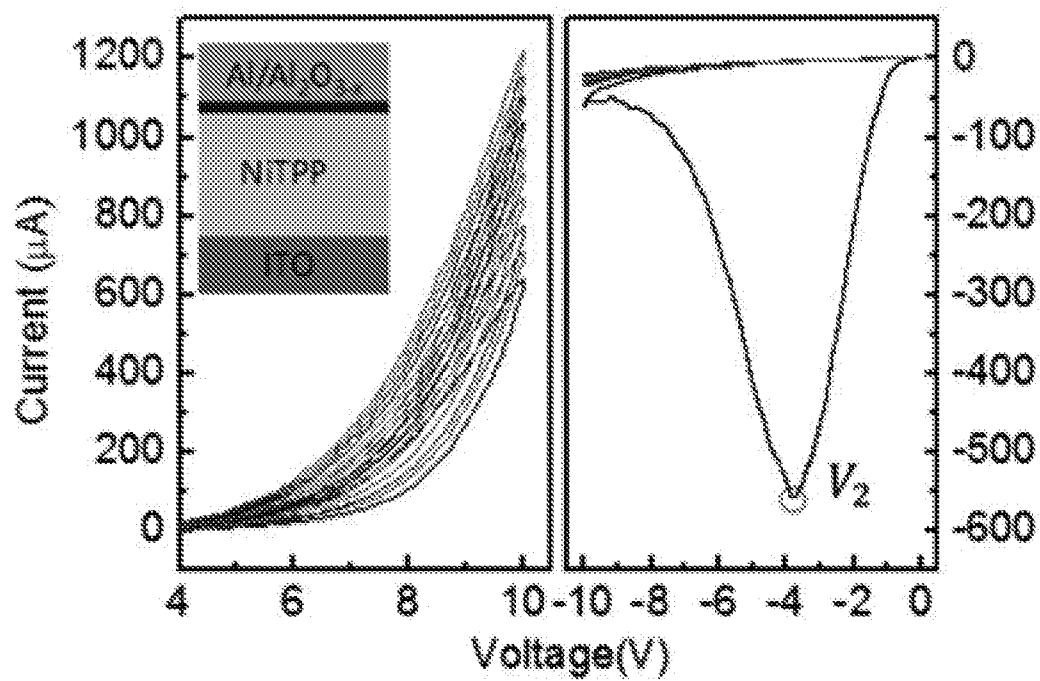


Fig.9

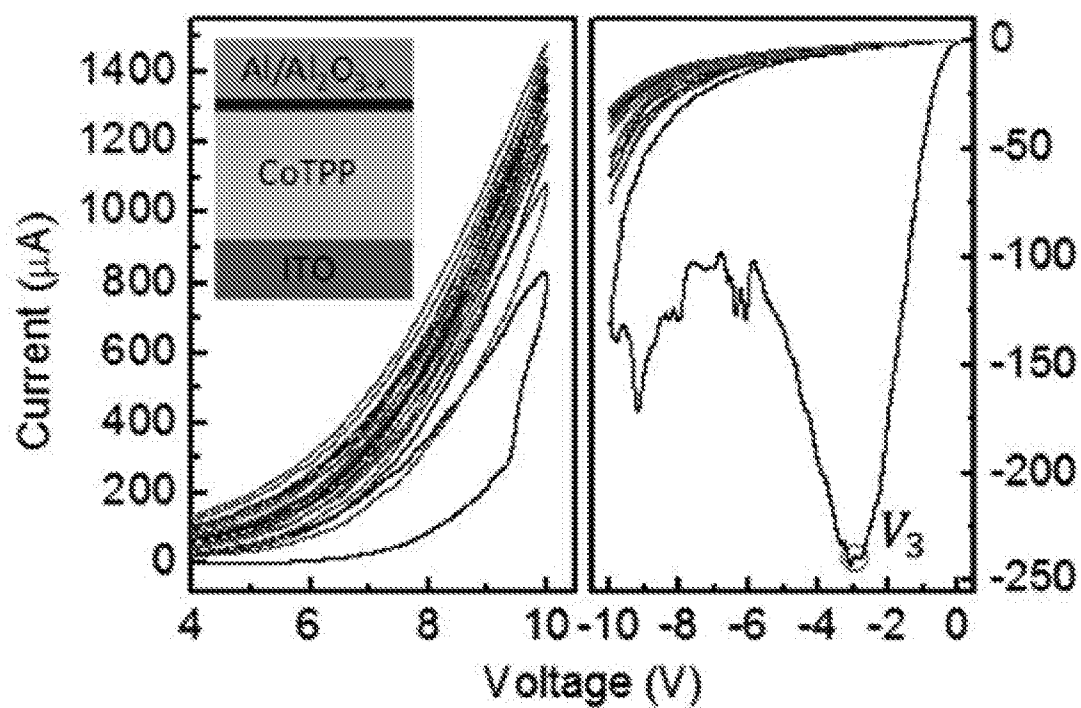


Fig.10

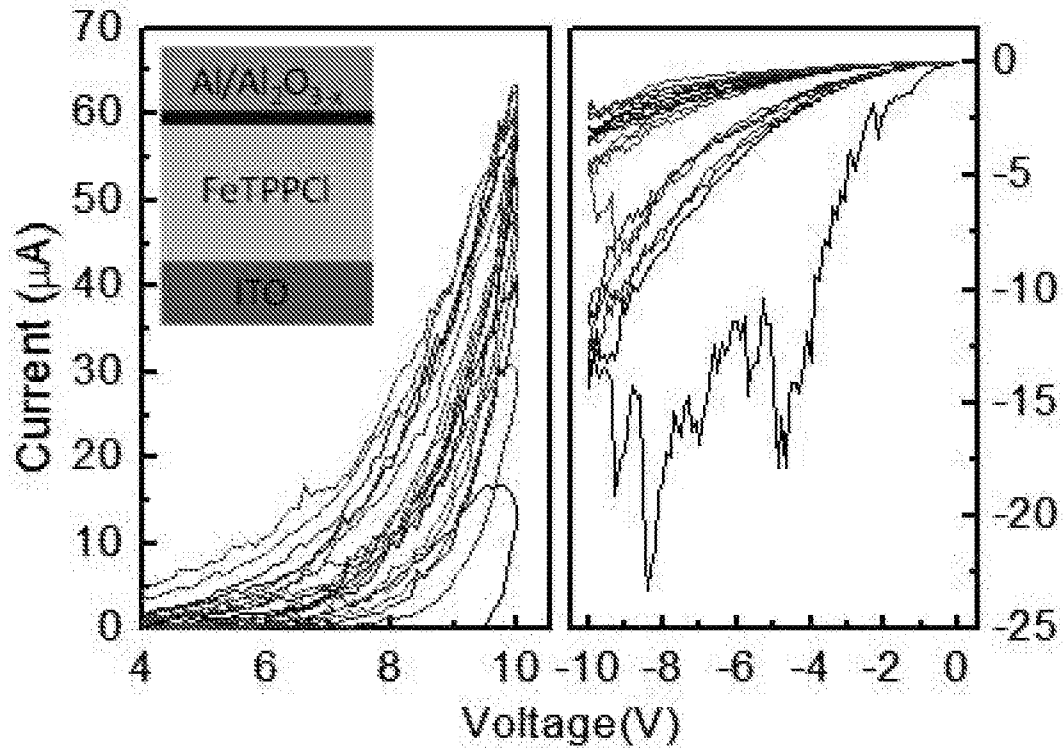


Fig.11

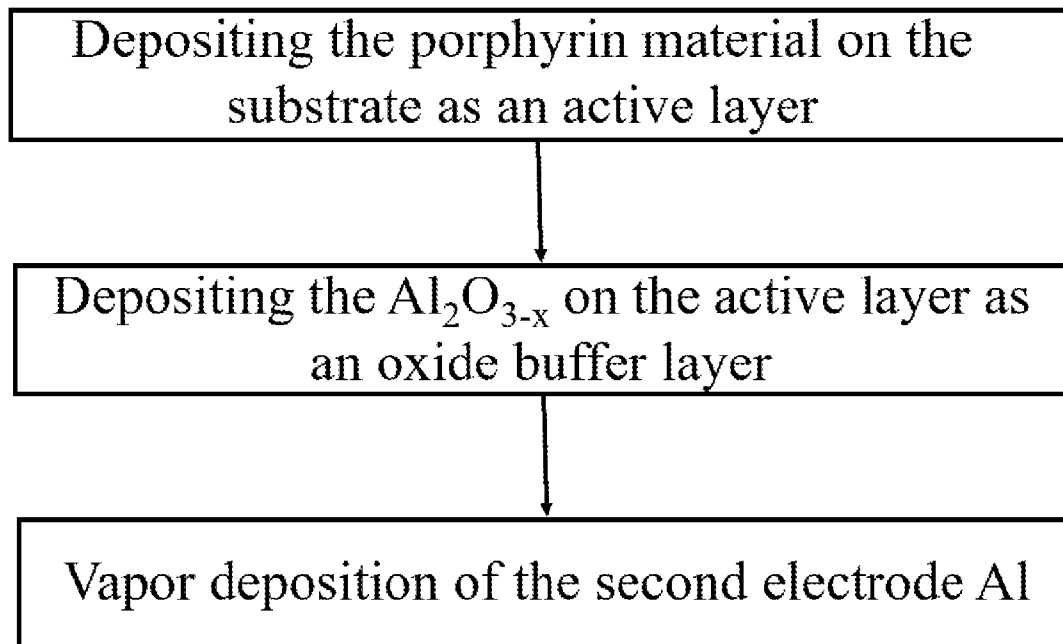


Fig.12

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2017/079886

A. CLASSIFICATION OF SUBJECT MATTER		
H01L 45/00(2006.01)i		
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)		
H01L		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
DWPI, CNTXT, CNABS, CNKI: memristor, porphyrin, anode, cathode, oxide, metal, TPP, ITO, aluminum, aluminium		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CN 102931346 A (INST MICROELECTRONICS CHINESE ACAD SCI) 13 February 2013 (2013-02-13) the whole document	1-13
A	CN 104979472 A (NINGBO INST MATERIAL TECHNOLOGY & ENG) 14 October 2015 (2015-10-14) the whole document	1-13
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A	US 2013343114 A1 (CARTER) 26 December 2013 (2013-12-26) the whole document	1-13
A	WO 2015034494 A1 (HEWLETT-PACKARD DEVELOPMENT COMPANY, L.P.) 12 March 2015 (2015-03-12) the whole document	1-13
☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
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Date of the actual completion of the international search		Date of mailing of the international search report
14 August 2017		24 August 2017
Name and mailing address of the ISA/CN		Authorized officer
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Facsimile No. (86-10)62019451		Telephone No. (86-10)62411328

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/CN2017/079886

Patent document cited in search report			Publication date (day/month/year)	Patent family member(s)			Publication date (day/month/year)
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