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(54) Title: A METHOD OF FORMING OXIDE QUANTUM DOTS AND USES THEREOF

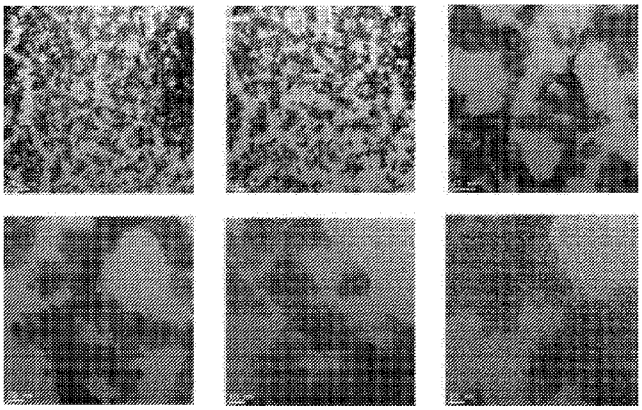

Fig. 2

(57) Abstract: A method of forming an oxide quantum dot precursor is disclosed. The method comprises preparing a sol by dissolving a precursor material for forming quantum dots in a liquid and aging the sol for a sufficient time period to form a network of substantially interlinked very small quantum dots. In some forms the aging is performed at a temperature greater than 60 degrees C for an hour or more. In some forms the aging is performed at a temperature greater than 85 degrees C for an hour or more.

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A METHOD OF FORMING OXIDE QUANTUM DOTS AND USES THEREOF

Technical Field

A method of forming oxide quantum dots is disclosed. A method of forming a transparent conductive oxide film on a substrate is also disclosed.

Background Art

Some materials, when quite small, have been known to exhibit new properties, such as quantum effects. Usually, these materials will be less than about 10 nanometers and may be referred to as 'quantum dots'. Such quantum dots can be formed as thin films deposited onto a substrate by a number of techniques including physical vapour deposition (PVD), chemical vapour deposition (CVD) and chemical synthesis.

Tin doped indium oxide (ITO) films have been prepared by a variety of methods, such as RF sputtering, reactive evaporation, chemical vapor deposition, and the sol-gel process. Among those, the sol-gel process is very cost-effective for the film preparation in large scale due to its easy control of the doping level, solution concentration and homogeneity without using expensive and complicated equipment when compared with other methods.

A challenge for the ITO thin film fabrication with sol-gel processing is the requirement to complex agents for the formation of stable sol solution and also the desirable viscosity. This not only increases the fabrication cost but also causes some pores and pinholes in the films, thus decreasing the electric conductivity significantly. As a result, it requires coating the films for many times to obtain the desired conductivity. However, the film coated with such a material usually has very low transparency.

The above references to the background art do not constitute an admission that the art forms a part of the common general knowledge of a person of ordinary skill in the art. The above references are also not intended to limit the application of the methods, substrate and use of a substrate as disclosed herein.

Summary

According to a first aspect, a method of forming an oxide quantum dot precursor is disclosed. The method comprises preparing a sol by dissolving a precursor

material for forming quantum dots in a liquid and aging the sol for a sufficient time period to form a network of substantially interlinked very small quantum dots. In some forms the aging is performed at a temperature greater than 60 degrees C for an hour or more. In some forms the aging is performed at a temperature greater than 85 degrees C for an hour or more.

According to a second aspect, a method of forming a thin film having an increased conductivity is disclosed. The method comprises preparing a sol by dissolving a precursor material for forming quantum dots in a liquid and aging the sol for a sufficient time period to form a network of substantially interlinked very small quantum dots, and depositing the sol on a substrate.

The current method in some forms comprises providing precursor materials for forming oxide quantum dots and dissolving the precursor materials in liquid. Nucleation of the oxide quantum dots is promoted in the liquid. The resultant sol is aged for a time sufficient to promote the formation of very small quantum dots which are substantially weakly interlinked to form an overall network between the quantum dots thus improving the conductivity of the sol and of a deposited film of the sol.

In some forms, the resultant sol when deposited on a substrate, forms a thin film having a useful level of conductivity. The resultant quantum dot precursors may be suitable for conductive applications, for deposition and for printing.

Various oxide quantum dots may be formed, such as indium tin oxide (ITO), fluorine-doped tin oxide (FTO), aluminium-doped zinc oxide (AZO), boron-doped zinc oxide (BZO), strontium ruthenium oxide (SRO), and some conductive polymers. As will be understood by those skilled in the art, various precursors may be used to achieve the required quantum dots. For example, the precursor materials may contain $SnCl_2$ and $In(NO_3)_3$ for forming ITO, $SnCl_2 \cdot H_2O$ and NH_4F for forming FTO, $AlCl_3$ and $Zn(CH_3COO)_2 \cdot 2H_2O$ for forming AZO, and $B(OCH_3)_3$ and $Zn(CH_3COO)_2 \cdot 2H_2O$ for forming BZO. Although, it should be appreciated that other precursor materials may be employed to obtain the same oxides. For example, $SnCl_4$ may be substituted for $SnCl_2$ and ITO will still be formed, or $Al(NO_3)_3$ or Al(O-i-Pr)$_3$ (Al-isopropoxide) may be substituted for $AlCl_3$ and AZO will still be formed. Similarly, substitutions to the precursors identified here are also known, and envisaged. For simplicity purposes,

further reference to oxide quantum dots, and precursors for forming the oxide quantum dots, will be made with respect to $SnCl_2$, or $SnCl_4$, and $In(NO_3)_3$ for forming ITO.

Brief Description

Notwithstanding any other forms that may fall within the scope of methods, substrate and use thereof as set forth in the Summary, specific embodiments will now be described, by way of example only, with reference to the accompanying drawings in which:

Figure 1 shows a schematic illustration of an embodiment of a growth mechanism of quantum dots;

Figure 2 shows TEM images of quantum dot precursor solution of one embodiment of the disclosure;

Figure 3 shows TEM images of quantum dot precursor solution of a second embodiment of the disclosure;

Figure 4 shows a solution aged for 1 hour at 40 degrees C;

Figure 5 shows a solution aged for 1 hour at 60 degrees C;

Figure 6 shows a solution aged for 1 hour at 90 degrees C.

Detailed Description

In some forms, disclosed is a method of forming quantum dot precursor sol, the method comprising preparing a sol by dissolving a precursor material for forming quantum dots in a liquid and aging the sol for a sufficient time period to form a network of substantially interlinked very small quantum dots.

In some forms the sol is aged at room temperature for a time period greater than 48 hours. In some forms the time period is greater than five days. In some forms the time period is greater than several weeks to months.

The step of aging may have the benefit of providing small quantum dots that are weakly linked providing for a greater level of conductivity across the sol and across the surface of a substrate upon which the sol is deposited.

In some forms the sol is aged at a temperature above room temperature. In these forms the time period need not be as long as when the sol is aged at room temperature. In some forms the sol is aged at a temperature above 60 degrees C.

In some forms, the step of aging is sufficient that the very small quantum dots have a maximum diameter less than 10nm. In some forms the step of aging is sufficient that the very small quantum dots have an average diameter less than 10nm. In some forms the step of aging is sufficient that the very small quantum dots have a maximum diameter less than 7nm. In some forms the step of aging is sufficient that the very small quantum dots have an average diameter less than 7nm. In some forms the step of aging is sufficient that the very small quantum dots have an average diameter less than 5nm. In some forms the step of aging is sufficient that the very small quantum dots have an average diameter less than 3nm.

In some forms the precursor material comprises tin-doped indium oxide. In some forms the precursor material comprises indium nitrate hydrate and tin chloride hydrate. In some forms the cation concentration of the sol is approximately 0.1M.

In some forms ethylene glycol is added as a complex agent.

In some forms, the method produces a deposited thin film and further comprises the step of depositing the quantum dot precursor sol on a substrate to form a deposited film.

In some forms the step of depositing the quantum dot precursor sol comprises depositing more than one layer of the quantum dot precursor sol on the substrate.

In some forms the deposited film has a resistance of less than 5000 ohm-m. In some forms the deposited film has a resistance of less than 1000 ohm-m.

In some forms the step of depositing the quantum dot precursor on a substrate is performed by spin coating, inkjet printing, doctor blade/slot die coating or screen printing.

Referring now to Figure 1, a general schematic illustration of an embodiment of the growth mechanism of quantum dots, as disclosed herein, is shown. The schematic illustration shown in Figure 1 shows a first step 10 of adding precursors such as, for example, indium and tin precursors to a solvent or solvents such as, for example, ethylene glycol.

In the second step 11, the composition is then aged at a temperature higher than room temperature. In some forms the composition is aged at greater than 60 degrees C for an hour or more. In some forms the composition is aged at greater than 80 degrees C for an hour or more.

As shown, the next step 12 is coating the aged sol onto a substrate. The coating step may be performed by means of ink-jet printing, slot die or other process that coats a substrate.

In the fourth step 13, the coated substrate is then annealed or dried. This step moves the process to the final step 14 and forms a transparent conductive thin film.

In some forms, the quantum dots may be deposited onto the substrate in a specific configuration, such as a decoration, shape or pattern so that a transparent conductive oxide film may be formed in the specific configuration. In this regard, the quantum dots may be deposited onto the substrate to form a transparent conductive oxide film in the shape of a logo or message. In some forms, the film may be used in circumstances where fogging or icing occurs (e.g. windscreens). The conductive oxide film may be used to facilitate clearing of such fog or icing in these circumstances. Where the film is in the shape of a logo or message, as the condensation, fog or ice is being cleared, the message or logo may appear on the substrate.

Prior to the quantum dots being deposited onto the substrate, the substrate may be pre-treated to reduce its surface energy. For example, the surface of the substrate may be cleaned (e.g. by deionized water, ethanol, acetate, etc), or the substrate may be pre-treated by, for example, UV-irradiation. Reduction of the surface energy of the substrate is believed to enlarge the liquid-air interface (i.e. as the liquid spreads out on the substrate surface), which may result in a thinner, more uniform film being formed.

In some forms, the transparent conductive oxide film may be dried on the substrate at ambient conditions. In other forms, the transparent conductive oxide film may be dried by UV-irradiation. In some forms a wide assortment of materials can be used as the substrate. For example, in addition to glasses, other transparent materials such as polymers may be employed.

In some forms, the quantum dots may be further deposited onto the substrate to form a thicker transparent conductive oxide film. In this regard, multiple layers of the transparent conductive oxide film may be deposited onto the substrate. In some forms,

these layers may be deposited directly onto the first layer/film of transparent conductive oxide. In other forms, these layers may be deposited onto the first (or preceding) layer/film of transparent conductive oxide only after the preceding layer has been dried. For example, the drying techniques identified above may be employed between the deposition of layers. Thicker transparent conductive oxide films may be preferred if a higher conductivity is required, or if regions of different conductivity are required. In some forms, the quantum dots may be further deposited onto the substrate to form a second transparent conductive oxide film that is discrete from the first transparent conductive oxide film (e.g. as two separate conductive circuits). In the case of discrete films, the application of current may be varied between the two discrete films, or may be time delayed. This may allow a message, pattern, logo, etc to be displayed on the substrate as it is being de-fogged or de-iced, etc.

In some forms less than five layers of deposition are required to produce a conductive thing film. In some forms three or fewer layers of deposition are required to produce a thin film. The reduction in the number of layers required for a thin film to be conductive increases the transparency of that film. In some forms the aging of the sol to produce very small quantum dots which are interlinked results in fewer layers being required for conductivity and this results in an increased transparency.

In some forms, the quantum dots may be deposited using ink-jet printing, spray printing, screen-printing, bar spreading, dip-coating, contact coating, or non-contact coating. Such deposition techniques allow the quantum dots to be deposited on large scale substrates, without being limited to the size of the chamber that would otherwise be required using known CVD, PVD or chemical synthesis techniques. Additionally, such deposition techniques may provide a significant cost savings when compared with known CVD, PVD or chemical synthesis techniques.

It should be appreciated that many other forms, for depositing the quantum dots onto the substrate, are well within the knowledge of the skilled addressee, and thus form part of the methods available to employ the method disclosed herein, even if the deposition methods themselves are not explicitly herein defined.

In another embodiment, the quantum dots may be deposited onto the substrate as two isolated, or discrete, transparent conductive oxide films (e.g. as two separate conductive circuits).

Examples

Non-limiting Examples of the methods, substrate and the use of a substrate will now be described, with reference to the Figures.

Example 1 – Preparation of 10 wt% Sn doped In_2O_3

$SnCl_2$ and $In(NO_3)_3$ in a weight ratio of 10:90 were mixed and dissolved in deionized (DI) water (giving a molecular concentration of In^{3+} of 0.1M). Ethylene glycol (EG) was used as complex agent. The cation concentration was controlled on 0.1M.

The solution was aged. In one procedure the solution was aged at room temperature for one month. The resulting precursor particles are shown in Fig. 2. The resultant particles are composed of very small quantum dots.

In a second procedure the solution was aged at room temperature for six months. The resulting precursor particles are shown in Fig. 3. The resultant particles are composed of very small quantum dots.

As shown, increasing the aging time tends toward increased linkages between the particles to form an entire network throughout the sample.

Example 2 - Spin-coating of ITO precursor quantum dots

ITO films were deposited on a slide of glass substrate by spin coating. The substrates were cleaned with soap water, rinsed in distilled water and ethanol. After drying, the substrates were radiated by UV with different duration. 20 μL of solution was applied to cover the glass substrate. The three-layer coated films were put into the oven to dry for 1 hr at different temperatures. The as-deposited films were annealed at

the suitable temperature, for example at 500°C, for a suitable duration, for example for 1 hour, in the inert gas environment, such as Ar atmosphere.

Subsequently, we used spin-coating technique to fabricate the ITO thin solid films on the surface of glasses. As shown in Table 1, all the films prepared by spin-coating show much higher electric conductivity than most of the reported ITO films prepared by the conventional sol-gel process. The thicker film results in a lower resistance. This well agrees with the previous work. Furthermore, the conductive ITO films with 18 layers still get a great transmittance in visible light. It demonstrates the superior physical properties that the electric conductivity is higher while the transparency is not scarified.

Table 1: Effects of the layers and annealing time on the electrical properties of ITO films by spin-coating

Sample No.	Layers No.	Annealing time / h	Resistance /Ω
1	3	1	500
	6		200
	9		100
	12		70-120
	15		70-80
	18	2	50-80
	21		40
2	3	1	500-700
	6		200
	9		150-200

	12	2	100-150
	15		90-120
3	3	2	700-1k

Example 3 - Inkjet printing

Inkjet printing represents an effective way to control the size, shape, thickness and pattern of thin films. It has been widely utilized to fabricate the thin solid films for the applications including solar cells, transistors, light-Emitting devices and sensors etc. Herein, the inkjet printing technology provides us an opportunity to tailor the ITO films with lower electric resistivity in the thinner film.

The surface is cleaned with the aforementioned technique. The Indium (III) nitrate hydrate (In(NO)3 xH2O) and Tin (IV) chloride hydrate (SnCl4 xH2O) were dissolved into ethylene glycol for aging at room temperature for a suitable duration, for example 30 days. Then the solution (1.5 ml) was filled into a cartridge of the ink-jet printer for printing (e.g. 10 layers each time) by using a Fujifilm DMP 2381 printer. The as-printed films were dried in oven at a suitable temperature, for example 80oC for 30 minutes and then annealed at a suitable temperature, for example 500oC for 2 hours, to obtain the ITO films.

Table 2: Summary of resistance of different ITO films by ink-jet printing

Number of layers	Method	Annealing	Resistance
10	Inkjet printing	Yes	1MΩ
20			200-300kΩ
30			~5kΩ

Table 2 lists of the fabrication parameters and the corresponding electric properties of the thin solid films coated with ink-jet printing technology. From Table 2 it can be found that the resistance of ITO films of 10 layers is high. However, with the increase of layers, the resistance has been reduced significantly. Therefore, the resistance of ITO films prepared by inkjet printer can be reduced significantly if more layers are deposited.

Example 4 - Doctor blade/Slot Die Coating

Doctor blade and slot die coating are the widely used techniques for effectively producing thin films on large surface area. The ink developed by the inventors is also compatible with doctor blade/slot die printing.

The glass substrate (for example, the width of the glass is around 25 cm) was cleaned with soap water, rinsed in distilled water and ethanol and then put into oven for 2 hours for drying at 80°C. Afterward, the glass substrate was put into an UV chamber for radiation with 10 minutes. Subsequently doctor blade applicator was used for coating the films on the glass substrate. A certain amount of solution was filled into the doctor blade applicator uniformly for coating. The coated film was dried at a suitable temperature, for example 70°C for 2 hours and then annealed at a suitable temperature, for example 500°C for 1 hour, in inert gas environment such as Ar atmosphere etc.

Table 3 Summary of resistance of different ITO films by doctor blade printing

Number of layers	Method	Annealing	Resistance
1	Doctor blade	Yes	10 kΩ
2			~500Ω
3			<100Ω

Example 5 : Screen Printing

The glass substrate (width around 10 cm) was cleaned with soap water, rinsed in distilled water and ethanol and then put into oven to dry at 80°C for 2 hours. Subsequently the glass substrate was put into an UV chamber for cleaning with 10 minutes. A certain amount of solution was filled on the top surface of the printing screen. Then use the brush to coat the film slowly. The thin solid films with the desired patterns can also be coated by applying the screens with the corresponding patterns. The coated film was dried at a suitable temperature, for example 70°C for 2 hours and then annealed at the suitable temperature, for example 500°C for 1 hour, in an inert gas environment, such as Ar atmosphere.

Referring now to Figures 4 – 6, the effect of aging temperature on the production of indium tin oxide quantum dots is shown. Fig. 4 shows a sol aged at 40 degrees C for an hour. No quantum dots are formed. Fig. 5 shows a sol aged at 60 degrees C for one hour. Some quantum dots are formed. Fig. 6 shows a sol aged at 90 degrees C for one hour. Significant quantum dots are formed. This large network of quantum dots may be interlinked to improve the conductivity of the sol and of a deposited film of the sol.

In some forms the time period for the step of aging the sol is a month or more, even up to six months and the step occurs at room temperature. In some forms a long aging process can result in a sol that has greater stability over time. However in other forms the time period is significantly shorter and the aging occurs at a higher temperature such as for example 70-90 degrees C for an hour to two hours. In some forms a long aging process can result in a sol that has greater stability over time.

In some forms the solubility of the precursor materials such as $SnCl_2$, or $SnCl_4$, and $In(NO_3)_3$ in the liquid used effect the formation of the ITO in the sol. In some forms the liquid has a viscosity that allows it to be utilised as an ink.

In some forms the process of aging and/or the selection of liquid solvent and materials results in fewer materials being required to provide a sol of the desired qualities such as viscosity and concentration as well as conductivity.

It will be understood to persons skilled in the art that many other modifications may be made without departing from the spirit and scope of the methods, substrate and use of a substrate as disclosed herein.

In the claims which follow and in the preceding description, except where the context requires otherwise due to express language or necessary implication, the word "comprise" or variations thereof such as "comprises" or "comprising" is used in an inclusive sense, i.e. to specify the presence of the stated features but not to preclude the presence or addition of further features in various embodiments of the methods, substrate and use of a substrate as disclosed herein.

CLAIMS

1. A method of forming quantum dot precursor sol, the method comprising:
preparing a sol by dissolving a precursor material for forming quantum dots in a liquid; and,
aging the sol for a sufficient time period to form a network of substantially interlinked very small quantum dots.

2. A method as defined in claim 1, wherein the sol is aged at room temperature for a time period greater than 48 hours.

3. A method as defined in any one of the preceding claims wherein the time period is greater than five days.

4. A method as defined in claim 1, wherein the sol is aged at a temperature above room temperature.

5. A method as defined in claim 4, wherein the sol is aged at a temperature greater than 80 degrees C for a time period of one hour or more.

6. A method as defined in any one of the preceding claims, wherein the step of aging is sufficient that the very small quantum dots have a maximum diameter less than 10nm.

7. A method as defined in any one of the preceding claims, wherein the precursor material comprises tin-doped indium oxide.

8. A method as defined in any one of the preceding claims, wherein the precursor material comprises indium nitrate hydrate and tin chloride hydrate.

9. A method as defined in any one of the preceding claims, wherein ethylene glycol is added as a complex agent.

10. A method as defined in any one of the preceding claims, wherein the cation concentration of the sol is approximately 0.1M.

11. A method of forming a transparent conductive film, the method comprising preparing a quantum dot precursor sol as defined in any one of the preceding claims, and depositing the quantum dot precursor sol on a substrate to form a deposited film.

12. A method as defined in claim 11, wherein the step of depositing the quantum dot precursor sol comprises depositing more than one layer of the quantum dot precursor sol.

13. A method as defined in claim 11 or 12, wherein the step of depositing the quantum dot precursor sol comprises depositing fewer than 5 layers of the quantum dot sol.

14. A method as defined in any one of claims 11 - 13, wherein the deposited film has a resistance of less than 5000 ohm-m.

15. A method as defined in any one of claims 11 - 14, wherein the deposited film has a resistance of less than 1000 ohm-m.

16. A method as defined in any one of claims 11 - 15, wherein the step of depositing the quantum dot precursor on a substrate is performed by spin coating.

17. A method as defined in any one of claims 11 - 15, wherein the step of depositing the quantum dot precursor on a substrate is performed by inkjet printing.

18. A method as defined in any one of claims 11 - 15, wherein the step of depositing the quantum dot precursor on a substrate is performed by doctor blade/slot die coating.

19. A method as defined in any one of claims 11 - 15, wherein the step of depositing the quantum dot precursor on a substrate is performed by screen printing.

20. A transparent conductive film prepared by the method of any one of claims 11 – 19.

21. A transparent conductive film having a network of weakly interlinked quantum dots.

22. The film of claim 21, wherein the film is less than five layers thick.

23. The film of claim 21 or 22, wherein the quantum dots comprise tin-doped indium oxide.

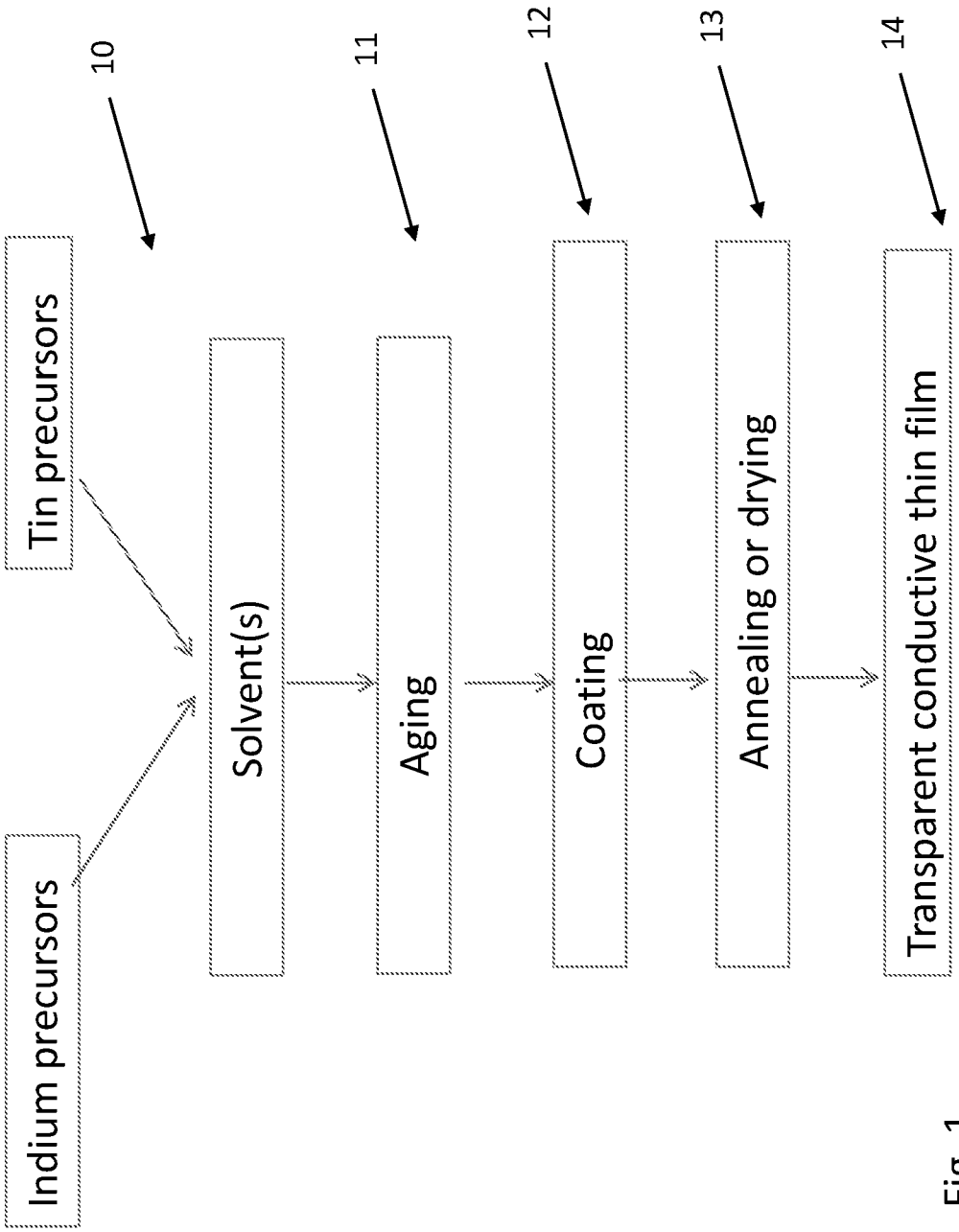
Tin precursors
Indium precursors
10
Solvent(s)
11
Aging
12
Coating
13
Annealing or drying
14
Transparent conductive thin film

Fig. 1

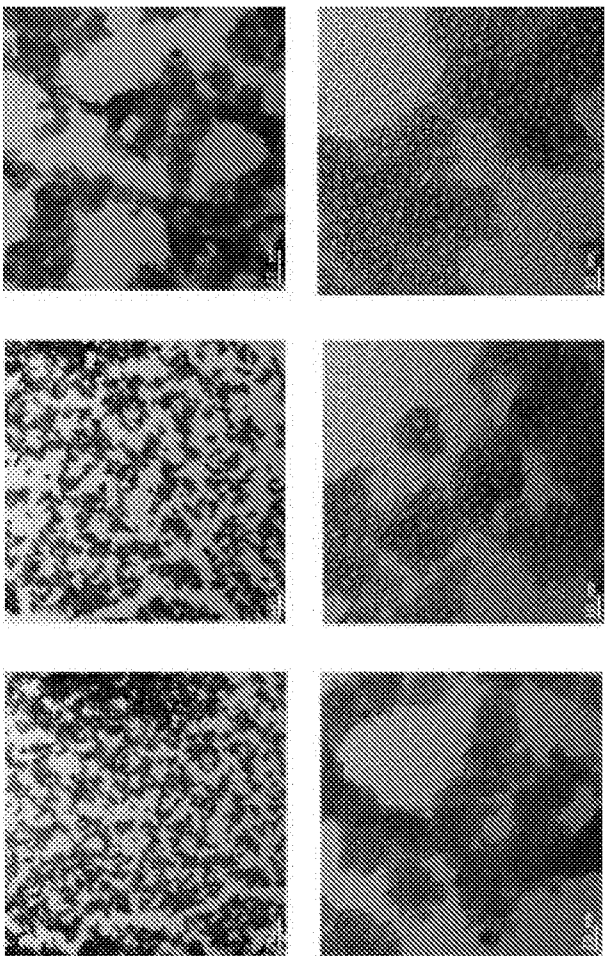

Fig. 2

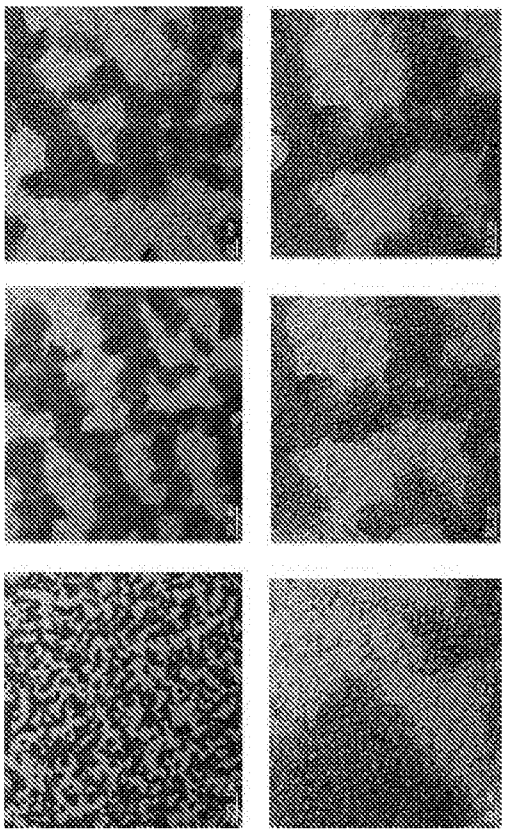

Fig. 3

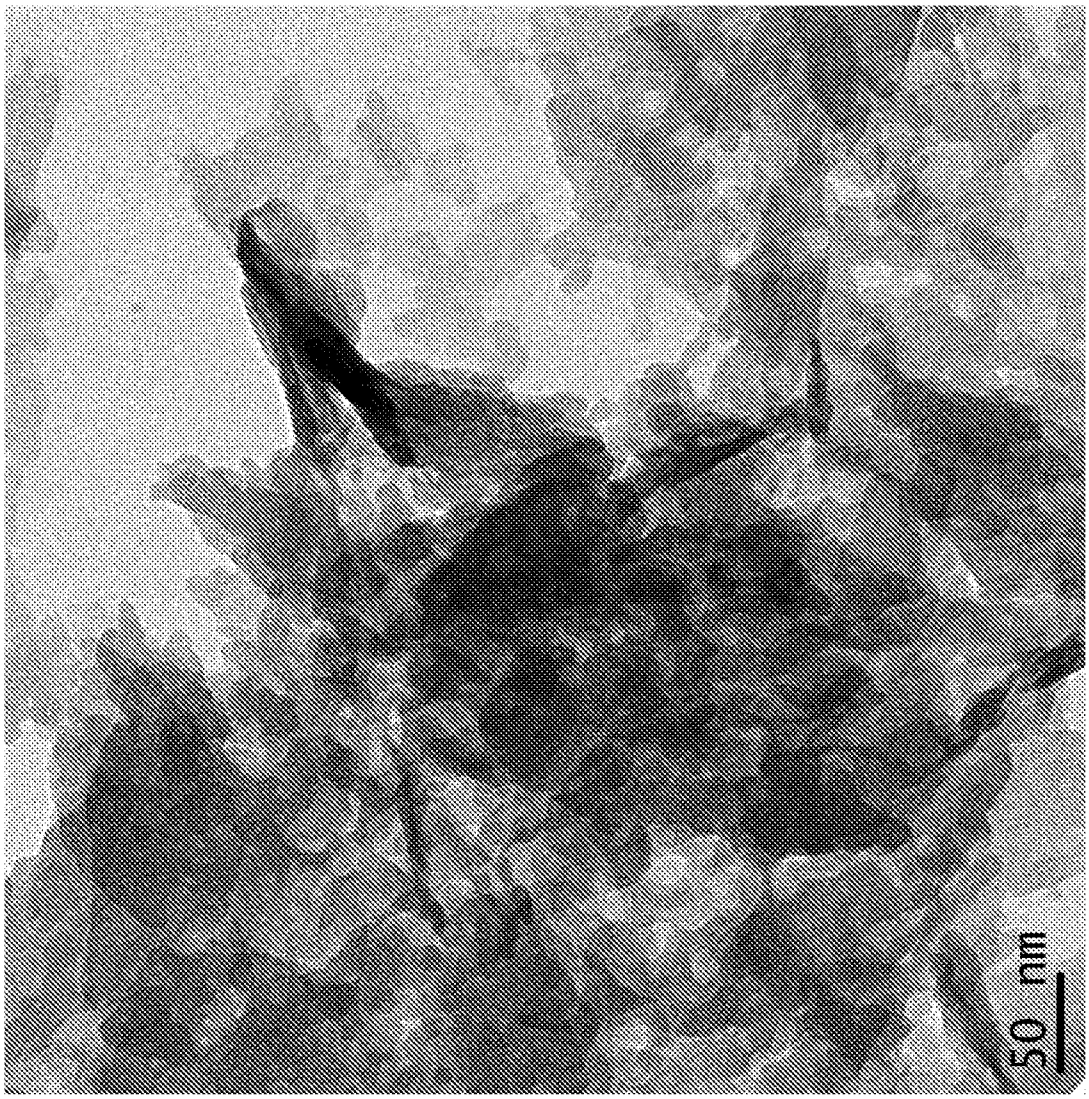

Fig. 4

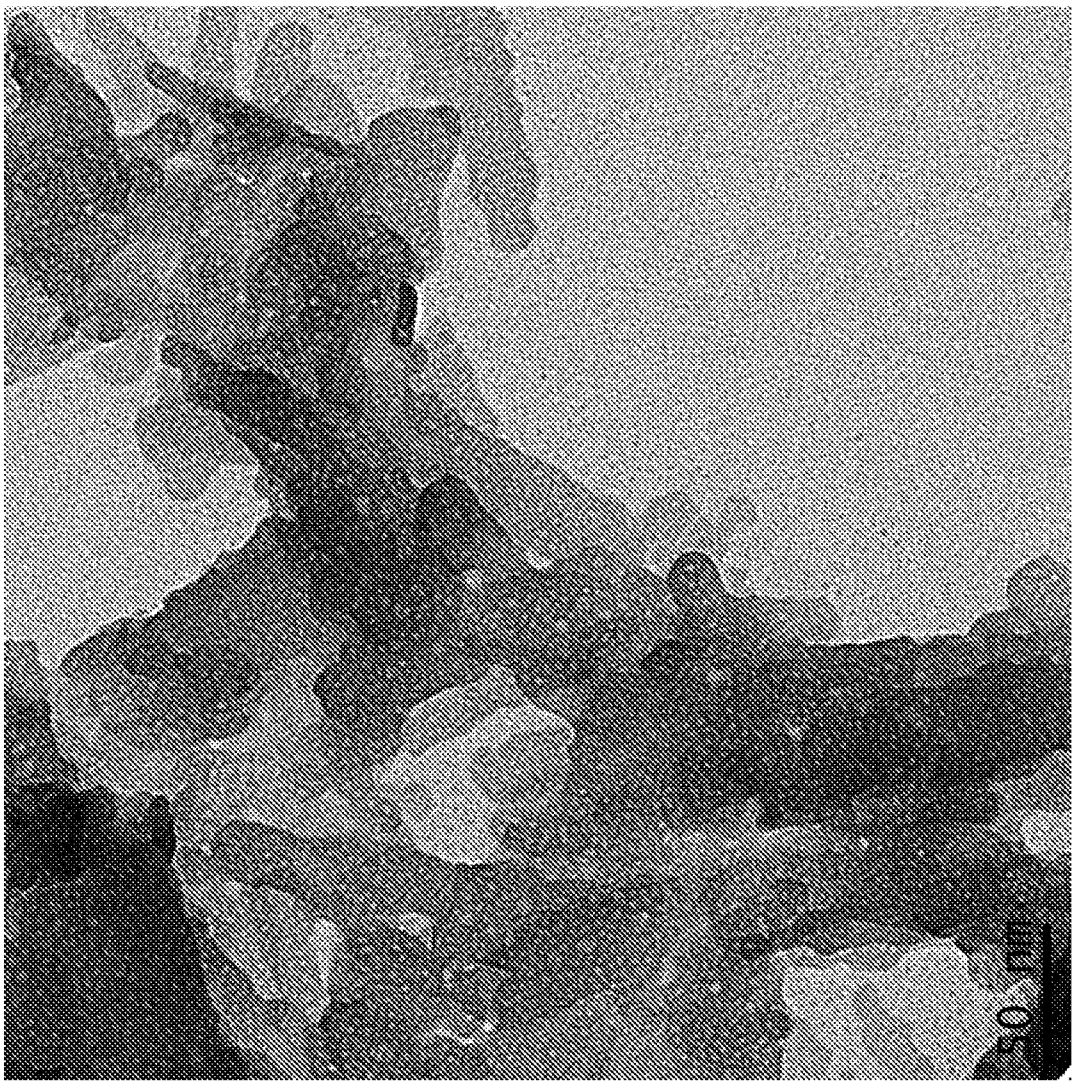

Fig. 5

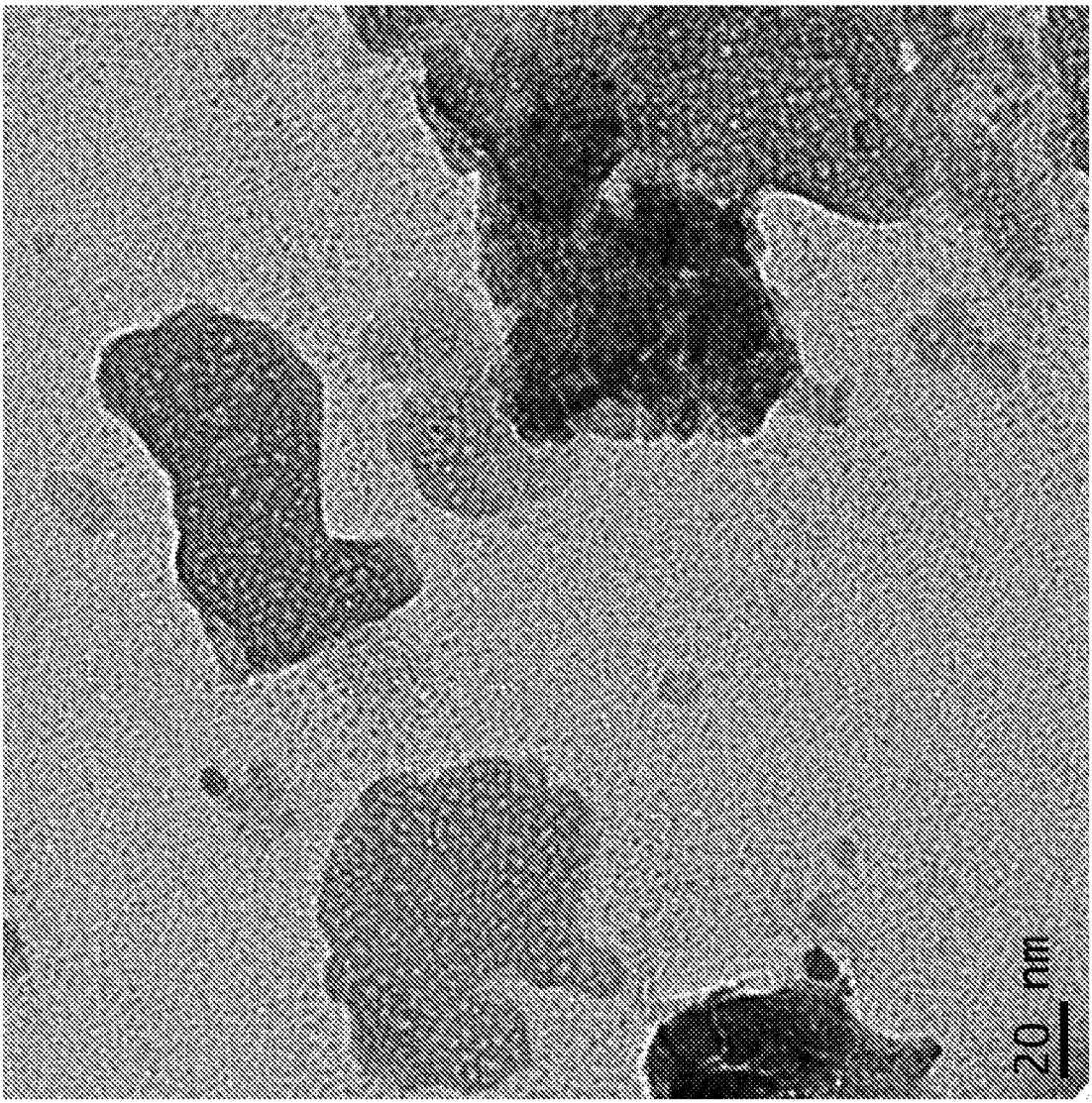

Fig. 6

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU2018/050685

A. CLASSIFICATION OF SUBJECT MATTER

C09K 11/66 (2006.01) C09K 11/62 (2006.01) H01L 31/0224 (2006.01) H01L 31/18 (2006.01) C01G 15/00 (2006.01) C01G 19/00 (2006.01)

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)

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)

PATENW, CAplus, INSPEC, COMPENDEX, CNFULL, KRFULL, JPFULL, ESPACENET, GOOGLE (PATENTS and SCHOLAR) with IPC/CPC: H01L31/022466/LOW, H01L31/1884/LOW, C09K11/661/LOW, C09K11/621/LOW, C01G15/00/LOW, C01G19/00/LOW and/or keywords: Quantum dot, precursor sol, aging, indium, tin, indium tin oxide, ITO, spin coat, inject print, doctor blade, slot die, screen print and similar terms searched.

Applicant: (Newsouth Innovations Pty Limited) and Inventor Names (LI, Sean Suixiang; CHU, Dewei) searched in CAplus, COMPENDEX, INSPEC, ESPACENET, GOOGLE (Patents & Scholar) and IP Australia internal databases.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
	Documents are listed in the continuation of Box C	

[X] Further documents are listed in the continuation of Box C [X] 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
30 July 2018	30 July 2018

Name and mailing address of the ISA/AU	**Authorised officer**
AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA Email address: pct@ipaustralia.gov.au	Luke Sweetman AUSTRALIAN PATENT OFFICE (ISO 9001 Quality Certified Service) Telephone No. +61262256163

INTERNATIONAL SEARCH REPORT		International application No.
C (Continuation).	DOCUMENTS CONSIDERED TO BE RELEVANT	**PCT/AU2018/050685**

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X	KUNDU S. et al., 'Synthesis and photoluminescence property of nanostructured sol-gel indium tin oxide on glass', Chemical Physics Letters, 2005, Vol. 414, Pages 107-110. Experimental and Figure 3	1, 6, 7-8, 11, 13, 16-23
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		KR 20090078099 A	17 Jul 2009
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Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.