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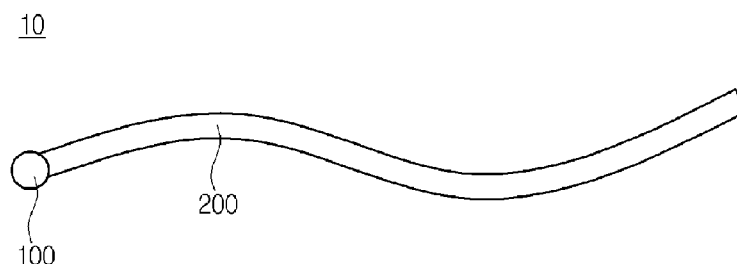
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(54) Title: METHOD OF FABRICATING NANO WIRE AND NANO WIRE COMPLEX

[Fig. 2]



(57) Abstract: Disclosed are a method of fabricating a nano wire and a nano wire complex. The method of fabricating a nano wire includes forming a plurality of seed particles by allowing a first ion to react with a second ion in a solvent, and forming a metallic nano wire by adding and heating a metallic compound in the solvent.



Description

Title of Invention: METHOD OF FABRICATING NANO WIRE AND NANO WIRE COMPLEX

Technical Field

- [1] The embodiment relates to a method of fabricating a nano wire and a nano wire complex.

Background Art

- [2] A transparent electrode including transparent material has been applied to various electronic products such as a display device, a solar cell, and a mobile device. Researches and studies on a nano wire, which has a wire-shape structure in a nano-meter size, as the transparent conductive material for the transparent electrode, have been actively carried out.
- [3] Since the nano wire has superior electrical conductivity, flexibility, and transmittance, the transparent electrode can represent superior characteristics. However, nano wires are easily aggregated during the reaction process, so that nano-particles are formed. Accordingly, the nano wire may not be easily fabricated. As a result, the product yield of the nano wire is significantly lowered to about 10%, so that the practical use of the nano wire may be difficult. Further, materials such as catalysts used to accelerate the reaction of forming the nano wire remain on the surface of the nano wire, so that the surface oxidation or the surface corrosion of the nano wire may occur, or the electrical conductivity may be degraded.

Disclosure of Invention

Technical Problem

- [4] The embodiment provides a long thin wire.

Solution to Problem

- [5] A method of fabricating a nano wire according to the embodiment includes forming a plurality of seed particles by allowing a first ion to react with a second ion in a solvent, and forming a metallic nano wire by adding and heating a metallic compound in the solvent.
- [6] According to the embodiment, the first ion may be a metallic ion, and the second ion may be a halogen ion.
- [7] According to the embodiment, the seed particles may include a metal equal to a metal constituting the metallic compound.
- [8] According to the embodiment, the seed particles and the metallic compound may include silver.
- [9] According to the embodiment, the seed particles may include silver chloride.

[10] According to the embodiment, a nano wire complex includes a metallic nano wire, and a seed particle bonded to the metallic nano wire. The seed particle has a diameter in a range of 5nm to 100nm.

[11] According to the embodiment, the seed particle is provided in the metallic nano wire or provided at one end of the metallic nano wire.

Advantageous Effects of Invention

[12] As described above, according to the method of fabricating the nano wire of the embodiment, after forming the seed particles from the solvent, the metallic nano wire is formed by using the seed particles. In this case, according to the method of fabricating the nano wire of the embodiment, the diameter of the seed particles can be properly adjusted. For example, the seed particles may have a very small diameter of about 5nm to about 100nm.

[13] In this case, the metallic nano wire may be grown from the seed particles. Since the seed particles have a very small diameter, the metallic nano wire may have very thin diameter.

Brief Description of Drawings

[14] FIG. 1 is a block diagram showing a method of fabricating a nano wire according to the embodiment;

[15] FIG. 2 is a view showing a nano wire complex according to the embodiment; and

[16] FIG. 3 is a view showing another example of a nano wire complex.

Mode for the Invention

[17] A method of fabricating a nano wire of the embodiment includes a step of forming a plurality of seed particles by allowing first ions to react to second ions in a solvent and a step of forming a metallic nano wire by adding a metallic compound to the solvent and heating the solvent having the metallic compound added thereto.

[18] In addition, the first ions may include metallic ions, and the second ions may include halogen ions.

[19] In addition, the seed particles and the metallic compound may include the same metal.

[20] Further, the seed particles and the metallic compound may include silver (Ag).

[21] In addition, the seed particles may include silver chloride (AgCl).

[22] Besides, each seed particle may have a diameter of about 5nm to about 100nm.

[23] Hereinafter, the disclosure will be described in detail with reference to accompanying drawings.

[24] FIG. 1 is a flowchart chart showing the method of fabricating the nano wire according to the embodiment.

[25] Referring to FIG. 1, a method for manufacturing a nano wire according to the

disclosure may include a step of heating a solvent (step ST10), a step of adding a capping agent to the solvent (step ST20), a step of forming a plurality of seed particles in the solvent (step ST30), a step of adding a fourth metallic compound to the solvent (step ST40), a step of adding a room-temperature solvent to the solvent (step ST50), and a step of refining a nano wire (step ST60). The steps are not essential steps, parts of the steps may not be performed according to the manufacturing method, and the sequence of the steps may be changed. Hereinafter, each step will be described in more detail.

- [26] According to the step of heating a solvent (step ST10), the solvent is heated at the reaction temperature suitable for forming the metallic nano wire.
- [27] The solvent may include polyol. The polyol serves as a mild reducing agent while serving as a solvent of mixing different materials, so that the polyol helps the formation of the metallic nano wire. For example, the polyol may include ethylene glycol (EG), propylene glycol (PG), glycerine, glycerol, or glucose. The reaction temperature may be variously adjusted by taking the types and the characteristics of solvents and the metallic compounds into consideration.
- [28] For example, if a silver nano wire is formed by using propylene glycol (PG) representing superior reduction power as a solvent, the reaction temperature may be in the range of about 80°C to 140°C. If the reaction temperature is less than 80°C, the reaction speed is reduced, so that reaction may not smoothly occur, and the fabricating time may be increased. If the reaction temperature exceeds 140°C, the silver nano wire may not be formed due to the aggregation phenomenon, and the product yield may be degraded.
- [29] As described above, according to the present embodiment, the silver nano wire may be fabricated at a reaction temperature lower than the reaction temperature (e.g., 160°C) according to the related art by using propylene glycol (PG) representing superior reduction power. According to the related art, since the reaction temperature is high, silver nano wires having a short length (e.g., less than 5 μ m), which is disadvantageous when forming a network structure, may be formed, and the product yield of the silver nano wires may be lowered. In contrast, according to the present embodiment, silver nano wires having a length of 20 μ m or more can be fabricated at a high product yield by reducing the reaction temperature.
- [30] Thereafter, according to the step of adding the capping agent to the solvent (step ST20), the capping agent inducing the forming of the wire is added to the solvent. If reduction for the forming of the nano wire is rapidly performed, metals are aggregated, so that the forming of the nano wire may be difficult. Accordingly, the capping agent prevents the metals from being aggregated by properly dispersing materials contained in the solvent.

- [31] The capping agent may include various materials. For example, the capping agent may include material selected from the group consisting of polyvinylpyrrolidone (PVP), polyvinyl alcohol (PVA), cetyl trimethyl ammonium bromide (CTAB), cetyl trimethyl ammonium chloride (CTAC), and polyacrylamide (PAA).
- [32] Then, according to the step of forming the seed particles in the solvent (step ST30), first and second metallic compounds are added to the solvent. Accordingly, first ions contained in the first metallic compounds may react to second ions contained in the second metallic compounds, thereby forming the seed particles. In this case, the first ions may react to the second ions to form a third metallic compound, and the seed particles may include the third metallic compound.
- [33] The first ions may include metallic ions. In more detail, the first ions may include gold ions, silver ions, platinum ions, or palladium ions
- [34] The second ions may include halogen ions. In more detail, the second ions may include chlorine ions, bromide ions, or iodide ions.
- [35] In other words, the third metallic compound contained in the seed particles may be expressed in following chemical formula 1.
- [36] Chemical Formula 1
- [37] MX
- [38] In this case, X represents Cl, Br, or I, and M represents Au, Ag, Pt, or Pd.
- [39] The third metallic compound may have very low solubility with respect to the solvent. Accordingly, the third metallic compound is extruded from the solvent and constitutes the seed particles.
- [40] The seed particles may have a very small diameter. The seed particles may have the diameter in the range of about 1nm to about 1 μ m. In more detail, the seed particles may have the diameter in the range of about 5nm to about 100nm. The above seed particles may be uniformly dispersed in the solvent.
- [41] The molar ratio of the second metallic compound to the first metallic compound may be about 1:1. In addition, the first metallic compound may be added to the solvent with the content of about 0.0001wt% to about 0.3wt%. Further, the second metallic compound may be added to the solvent with the content of about 0.0001wt% to about 0.3wt%.
- [42] The first metallic compound may include salts including the first ions. In addition, the first metallic compound may include nitrates. In more detail, the first metallic compound may include silver nitrate.
- [43] The second metallic compound may include salts including the second ions. In addition, the second metallic compound may include sodium salts. In more detail, the second metallic compound may include sodium chloride.
- [44] Thereafter, in the step of adding a fourth metallic compound to the solvent (step

ST40), a reaction solution is prepared by adding the fourth metallic compound to the solvent.

- [45] In this case, the fourth metallic compound melt in an additional solvent may be added to a solvent having the capping agent added thereto and the seed particles provided therein. The additional solvent may include the same material as that constituting an initial solvent or a material different from that constituting the initial solvent. In addition, the fourth metallic compound may be added after a predetermined time elapses from time at which the seed particles are formed. Accordingly, the temperature can be stabilized to a proper reaction temperature.
- [46] In this case, the fourth metallic compound may include metal used to form a metallic nano wire to be fabricated. In order to form silver nano wire, the metallic compound may include AgNO_3 , or $\text{KAg}(\text{CN})_2$.
- [47] As described above, if the fourth metallic compound is added to the solvent including the capping agent and the seed particles, reaction occurs so that the fabrication of the metallic nano wire is started. In this case, the metallic nano wire may be grown from the seed particles. In other words, metal extruded by reducing the fourth metallic compound is grown from each seed particle to form the metallic nano wire.
- [48] In this case, since the seed particles have a very small diameter, the metallic nano wire may be grown with a small diameter.
- [49] After the metallic nano wire has been completely grown, the seed particles may be removed through the following processes such as a refining process. In other words, in the following processes, the seed particles may be separated from the metallic nano wire and removed.
- [50] However, a portion of the seed particles may remain. Accordingly, a portion of the third metallic compound may be detected from the metallic nano wire according to the present embodiment.
- [51] In other words, the seed particles may be bonded with the metallic nano wire thereby forming a nano wire complex.
- [52] FIG. 2 is a view showing the nano wire complex according to the embodiment. FIG. 3 is a view showing another nano wire complex.
- [53] As shown in FIGS. 2 and 3, portions of the metallic nano wire may have the form a nano wire complex 10 or 11. The seed particles 100 are bonded with the metallic nano wire 200. The ratio of the metallic nano wire representing the form of the nano wire complex 10 or 11 may be about 0.1% to about 0.001%.
- [54] In particular, as shown in FIG. 2, the seed particles 100 may be provided at one end of the metallic nano wire 200. In addition, as shown in FIG. 3, seed particles 110 may be provided in the metallic nano wire 200.
- [55] In this case, as described above, the diameters of the seed particles 100 and 110 may

be in the range of about 1nm to about 1 μ m, in more detail, the range of about 5nm to about 100nm. In more detail, the diameters of the seed particles 110 and 110 may be in the range of about 10nm to about 50nm. As described above, when the seed particles 100 and 110 having a very small diameter are detected, the metallic nano wire having a small diameter is formed through the fabricating method according to the present embodiment.

- [56] According to the present embodiment, 60 weight part to 330 weight part of the capping agent may be added based on 100 weight part of the metallic compound such as AgNO₃, or KAg(CN)₂. If less than 60 weight part of the capping agent is added, the aggregation phenomenon can be sufficiently prevented. If over 330 weight part of the capping agent is added, metallic nano particles having a spherical shape or a cubic shape may be formed, and the capping agent remains in the fabricated metallic nano wire, so that the electrical conductivity of the metallic nano wire may be degraded.
- [57] In addition, the first and second metallic compounds may have the content in the range of 0.00001 weight part to 0.5 weight part based on 100 weight part of the fourth metallic compound. If less than 0.00001 weight part of the first and second metallic compounds is added, the reaction may not be sufficiently accelerated. In addition, if over 0.5 weight part of the first and second metallic compounds is added, silver is rapidly reduced so that silver nano particles are generated or the nano wire may have a thick diameter and a short length. In addition, catalyst remains in the metallic nano wire so that the electrical conductivity may be degraded.
- [58] Thereafter, according to the step of adding the room-temperature solvent to the solvent (step ST50), the room-temperature solvent is added to the solvent in which reaction is started. The room-temperature solvent may include a material identical to or different from a material used in the initial stage. For example, the room-temperature solvent may include polyol such as ethylene glycol and propylene glycol.
- [59] As the solvent, in which the reaction is started, is continuously heated in order to maintain the constant reaction temperature, the temperature may be increased in the process of the reaction. As described above, the reaction temperature may be more constantly maintained by temporarily degrading the temperature of the solvent by adding the room-temperature solvent to the solvent in which the reaction is started.
- [60] The step of adding the room-temperature solvent (step ST50) may be performed one time or several times by taking the reaction time, and the temperature of the reaction solution into consideration.
- [61] Thereafter, in the step of refining the nano wire (step ST60), the metallic nano wire is refined and collected in the reaction solution.
- [62] In more detail, if acetone serving as a non-polar solvent is added to the reaction solution instead of water, the metallic nano wire is deposited at the lower portion of the

solution due to the capping agent remaining on the surface of the metallic nano wire. This is because the capping agent is not dissolved in the acetone, but aggregated and deposited although the capping agent is sufficiently dissolved in the solvent. Thereafter, when the upper portion of the solution is discarded, a portion of the capping agent and nano particles are discarded.

[63] If distill water is added to the remaining solution, metallic nano wire and metallic nano particles are dispersed. In addition, if acetone is more added, the metallic nano wire is deposited, and the metallic nano particles are dispersed in the upper portion of the solution. Thereafter, if the upper portion of the solution is discarded, a part of the capping agent and the aggregated metallic nano particles are discarded. After collecting the metallic nano wire by repeatedly performing the above processes, the metallic nano wire is stored in the distill water. The metallic nano wire can be prevented from being re-aggregated by storing the metallic nano wire into the distill water.

[64] As described above, according to the method of fabricating the metallic nano wire of the embodiment, the metallic nano wire is grown by using seed particles having a very small diameter. Accordingly, the metallic nano wire having a small diameter can be formed.

[65]

[66] Experimental Example

[67] 200ml of propylene glycol was heated at a temperature of 126°C, and 6.7g of polyvinylpyrrolidone and 0.1g of potassium bromide were added and melted. Thereafter, 0.35mmol of sodium salt and 0.35mmol of AgNO₃ were added to form the seed particles. After about 10mins were elapsed, 2.3g of AgNO₃ was melted in 100ml of propylene glycol and added to a solution containing the polyvinylpyrrolidone and the seed particles. Then, the reaction was continued for about 2 hours, so that the fabrication of the silver nano wire was finished.

[68] After the solution, which had been subject to the reaction, was diluted by using 500ml of acetone, 600ml of acetone was added to the diluted solution. Then, the upper portion of the solution having propylene glycol, and silver nano particles dispersed therein was discarded. After repeatedly performing the above processes three times, the result was stored in 10ml of distill water.

[69]

[70] Comparative Example

[71] Different from the experimental example, silver chloride particles having the average diameter of about 2.5 μ m were added to the solvent instead of forming seed particles through the reaction between sodium salt and silver nitrate. Remaining procedures were performed in the same manner as that of the experimental example.

[72]

[73] Result

[74] As shown in Table 1, a thinner and longer silver nano wire was formed in the experimental example.

[75] Table 1

[Table 1]

	average diameter(nm)	average length(μm)
Experimental Example	45	22
Comparative Example	65	20

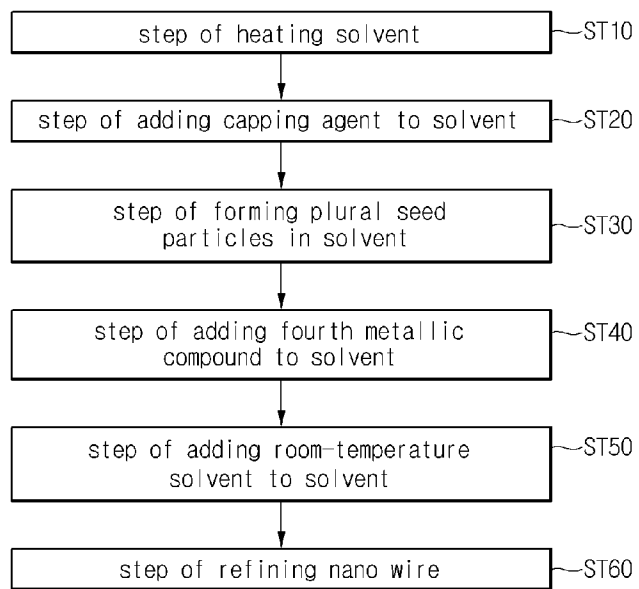
[76] Any reference in this specification to “one embodiment,” “an embodiment,” “example embodiment,” etc., means that a particular feature, structure, or characteristic described in connection with the embodiment is included in at least one embodiment of the invention. The appearances of such phrases in various places in the specification are not necessarily all referring to the same embodiment. Further, when a particular feature, structure, or characteristic is described in connection with any embodiment, it is submitted that it is within the purview of one skilled in the art to effect such feature, structure, or characteristic in connection with other ones of the embodiments.

[77] Although embodiments have been described with reference to a number of illustrative embodiments thereof, it should be understood that numerous other modifications and embodiments can be devised by those skilled in the art that will fall within the spirit and scope of the principles of this disclosure. More particularly, various variations and modifications are possible in the component parts and/or arrangements of the subject combination arrangement within the scope of the disclosure, the drawings and the appended claims. In addition to variations and modifications in the component parts and/or arrangements, alternative uses will also be apparent to those skilled in the art.

Claims

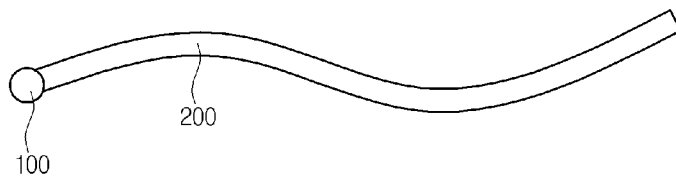
- [Claim 1] A method of fabricating a nano wire, the method comprising:
forming a plurality of seed particles by allowing a first ion to react with a second ion in a solvent; and
forming a metallic nano wire by adding and heating a metallic compound in the solvent.
- [Claim 2] The method of claim 1, wherein the seed particles have a diameter in a range of 5nm to 100nm.
- [Claim 3] The method of claim 1, wherein the first ion is a metallic ion, and the second ion is a halogen ion.
- [Claim 4] The method of claim 1, wherein the seed particles include a metal equal to a metal constituting the metallic compound.
- [Claim 5] The method of claim 1, wherein the seed particles and the metallic compound include silver.
- [Claim 6] The method of claim 1, wherein the seed particles include silver chloride.
- [Claim 7] A nano wire complex comprising:
a metallic nano wire; and
a seed particle bonded with the metallic nano wire,
wherein the seed particle has a diameter in a range of 5nm to 100nm.
- [Claim 8] The nano wire complex of claim 7, wherein the seed particle is provided in the metallic nano wire.
- [Claim 9] The nano wire complex of claim 7, wherein the seed particle is provided at one end of the metallic nano wire.
- [Claim 10] The nano wire complex of claim 7, wherein the seed particle includes chloride.
- [Claim 11] The nano wire complex of claim 10, wherein the metallic nano wire includes silver, and the seed particle includes silver chloride.

[Fig. 1]



[Fig. 2]

10



[Fig. 3]

11

