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(54) **UNIFORM ASPECT RATIO SILVER NANOWIRES PREPARATION METHOD**

VERFAHREN ZUR HERSTELLUNG VON SILBER-NANODRÄHTEN MIT EINHEITLICHEM
ASPEKTVERHÄLTNIS

PROCÉDÉ DE PRÉPARATION DE NANOFILS D'ARGENT À RAPPORT D'ASPECT UNIFORME

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Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

[0001] The present invention relates to a preparation method for silver nanowires, and more particularly, to a preparation method for silver nanowires with a uniform aspect ratio.

2. Description of Related Art

[0002] Based on the properties of high specific surface area, electrical conductivity and thermal conductivity, silver nanowires have a broad application prospect. Especially, the silver nanowires can be used as an alternative material of ITO in fields such as solar energy, OLED, and flexible and large-size touch screen displays, and used in fields such as conductive polymers, composite materials, and electrode printing ink additives. Therefore, the preparation of silver nanowires has become a focus of researchers. At present, there are many documents and patent reports on the preparation methods and applications of silver nanowires. For example, Xia Younan, the first one who prepares silver nanowires with a polyol method, proposed the preparation of silver nanowires with a high aspect ratio by using Pt as seeds and reducing silver nitrate by ethylene glycol (Advanced Materials (2002) 14,883). The polyol reduction method was combined with the hydrothermal method, and a small amount of sodium chloride, ferric chloride, copper chloride and other metal halide were introduced to function with Ag ions to form a colloid, which serves as seeds to prepare silver nanowires. The silver nanowires can be quickly prepared by the combination of the polyol method and the microwave method. However, the silver nanowires thus obtained have a relatively non-uniform aspect ratio, and have a large amount of impurities. For example, Chinese Patent Application No. 200810019828.6 disclosed a batch preparation method for silver nanowires, and Chinese Patent Application No. 201010559335.9 disclosed a method for preparing silver nanowires with controlled diameter by using a cation-controlled microwave method. However, he prepared silver nanowires are less than 30 μm in length, and have a non-uniform aspect ratio. Chinese Patent No. CN 1843670A reported the use of a composite solvent of glycerol and water or ethanol or isopropanol for reduction to prepare silver nanowires. The obtained silver nanowires have a length of 5-200 μm and a diameter of 70-90 nm. Furthermore, Chinese Patent CN 104785794 A discloses a preparation method for silver nanowires even in length-diameter ratio, comprising the following steps: dissolving silver nitrate in glycerol to get a solution A; dissolving polyvinylpyrrolidone in glycerol to get a solution B; mixing uniformly the solution A and the solution B to form a solution C; and finally, transferring the resulting solution into a reaction kettle for re-

action at 150°, and centrifuging the reactants to obtain silver nanowires.

[0003] Although many preparation methods for silver nanowires have been reported, few of them can obtain silver nanowires with a uniform aspect ratio and no other particles or impurities. Therefore, it is very important to invent a method that has a stable process, has fewer control factors, is simple and quick, can prepare a large quantity of Ag nanowires.

SUMMARY OF THE INVENTION

[0004] The present invention is directed to a preparation method of silver nanowires with a uniform aspect ratio, which is simple, easy to control, and cost-effective.

[0005] The present invention is achieved through the following technical solution.

[0006] A preparation method for silver nanowires includes the following steps:

- (1) dissolving silver nitrate in glycerol to get a solution A;
- (2) dissolving polyvinylpyrrolidone in glycerol to get a solution B;
- (3) mixing uniformly the solution A and the solution B to form a solution C; and
- (4) adding an ultrapure water medium into the solution C and mixing uniformly to form a solution D, finally, transferring the solution D into a reaction kettle and putting the kettle into an oven with a set temperature of 160°C, for reaction at 150°C $\pm$ 10°C, and centrifuging the reactants to obtain silver nanowires. The obtained silver nanowires have high purity and a uniform aspect ratio.

[0007] Preferably, reaction time in the reaction kettle is 9-10 hours.

[0008] To further improve purity and uniformity of the aspect ratio of the silver nanowires, in step (1), 0.16 g silver nitrate is dissolved at room temperature in 20 ml of glycerol to get the solution A.

[0009] In step (2), 5 g polyvinylpyrrolidone is dissolved at room temperature in 80 ml of glycerol to get the solution B.

[0010] In step (4), 5 ml of the ultrapure water is added into the solution C and mixed uniformly to form the solution D.

[0011] The centrifuging is performed twice.

[0012] The present invention has the following beneficial effects:

[0013] The preparation method of the present invention is simple to operate, easy to control, cost-effective, and very suitable for large-scale industrial production. The prepared silver nanowires have a uniform aspect ratio, and transparent conductive films based on the silver nanowires have a high transparency of 90-91%. The silver nanowires have a diameter of up to 30-40 nm and a length of 10-20 μm , and the silver nanowires with the

aspect ratio have small resistance, which is beneficial to the improvement of electrical conductivity. In addition, the silver nanowires have no particles and feature high purity.

BRIEF DESCRIPTION OF THE DRAWINGS

[0014]

FIG. 1 is a scanning electron microscope (SEM) diagram of a silver nanowire synthesized in Example 1 of the present invention; and

FIG. 2 is a diagram showing transmittance of a conductive thin film made of silver nanowires synthesized in Example 1 of the present invention.

DESCRIPTION OF THE EMBODIMENTS

[0015] The technology of the present invention will be further described below with reference to specific examples.

Example 1

[0016] 0.16 g silver nitrate was dissolved at room temperature in 20 ml of glycerol to get a solution A; 5 g polyvinylpyrrolidone was dissolved at room temperature in 80 ml of glycerol to get a solution B; the solution A and the solution B were mixed uniformly to form a solution C; then, 5 ml of ultrapure water was added into the solution C and mixed uniformly to form a solution D, finally, the solution D was transferred into a reaction kettle, the reaction kettle was put into an oven with a set temperature of 160°C, and the reaction ended after a certain time of reaction.

[0017] The Ag nanowire mother solution in the reaction kettle was diluted with alcohol and centrifuged twice to obtain precipitated silver nanowires having a diameter of 30-40 nm and a length of 10-20 μm, which were dispersed in isopropanol. FIG. 1 is a scanning electron microscope (SEM) diagram of a silver nanowire synthesized in Example 1.

[0018] The silver nanowires prepared in Example 1 above were formulated into 1g/L silver nanowire slurry, and 500 μL of slurry was uniformly coated on an A4-size transparent film to prepare a transparent conductive film. The sheet resistance of the transparent conductive film was 42 ohm/sq. Then, the transmittance of the transparent conductive film was measured with a UV-Vis spectrophotometer. As shown in FIG. 2, at the visible light wavelength of 550 nm, the transmittance of the transparent conductive film was 90.8%.

Comparative example 2

[0019] 0.16 g silver nitrate was dissolved at room temperature in 20 ml of glycerol to get a solution A; 5 g polyvinylpyrrolidone was dissolved at room temperature in

80 ml of glycerol to get a solution B; the solution A and the solution B were mixed uniformly to form a solution C; then, 2.5 ml of ultrapure water was added into the solution C and mixed uniformly to form a solution D, finally, the solution D was transferred into a reaction kettle, the reaction kettle was put into an oven with a set temperature of 160°C, and the reaction ended after a certain time of reaction.

[0020] The Ag nanowire mother solution in the reaction kettle was diluted with alcohol and centrifuged twice to obtain precipitated silver nanowires having a diameter of 30-40 nm and a length of 10-20 μm, which was dispersed in isopropanol.

15 Comparative example 3

[0021] 0.32 g silver nitrate was dissolved at room temperature in 20 ml of glycerol to get a solution A; 7 g polyvinylpyrrolidone was dissolved at room temperature in 80 ml of glycerol to get a solution B; the solution A and the solution B were mixed uniformly to form a solution C; then, 10 ml of ultrapure water was added into the solution C and mixed uniformly to form a solution D, finally, the solution D was transferred into a reaction kettle, the reaction kettle was put into an oven with a set temperature of 160°C, and the reaction ended after a certain time of reaction.

[0022] The Ag nanowire mother solution in the reaction kettle was diluted with alcohol and centrifuged twice to obtain precipitated silver nanowires having a diameter of 30-40 nm and a length of 10-20 μm, which was dispersed in isopropanol.

35 Claims

1. A preparation method for silver nanowires with a uniform aspect ratio, comprising the following steps:

- (1) dissolving 0.16 g silver nitrate at room temperature in 20 ml of glycerol to get a solution A;
- (2) dissolving 5 g polyvinylpyrrolidone at room temperature in 80 ml of glycerol to get a solution B;
- (3) mixing uniformly the solution A and the solution B to form a solution C; and
- (4) adding 5 ml of an ultrapure water into the solution C and mixing uniformly to form a solution D, finally, transferring the solution D into a reaction kettle, putting the reaction kettle into an oven with a set temperature of 160°C, and ending the reaction after a certain time of reaction; diluting a silver nanowire mother solution in the reaction kettle with alcohol and centrifuging twice to obtain precipitated silver nanowires having a diameter of 30-40 nm and a length of 10-20 μm, as determined using scanning electron microscopy.

2. The preparation method according to claim 1, where-
in reaction time in the reaction kettle is 9-10 hours.

Patentansprüche

1. Herstellungsverfahren von Silbernanodrähten mit
einem einheitlichen Seitenverhältnis, umfassend die
folgenden Schritte:

(1) Lösen von 0,16 g Silbernitrat bei Raumtem-
peratur in 20 ml Glycerin, um eine Lösung A zu
erhalten;

(2) Lösen von 5 g Polyvinylpyrrolidon bei Raum-
temperatur in 80 ml Glycerin, um eine Lösung
B zu erhalten;

(3) gleichmäßiges Mischen der Lösung A und
der Lösung B, um eine Lösung C zu erhalten;
und

(4) Zugabe von 5 ml ultrareinstem Wasser zu
der Lösung C und gleichmäßiges Mischen, um
eine Lösung D zu bilden, schließlich Überführen
der Lösung D in einen Reaktionskessel, Einset-
zen des Reaktionskessels in einen Ofen mit ei-
ner eingestellten Temperatur von 160°C und
Beenden der Reaktion nach einer bestimmten
Reaktionszeit; Verdünnen einer Silbernano-
draht-Stammlösung in dem Reaktionskessel mit
Alkohol und zweimaliges Zentrifugieren, um
ausgefällte Silbernanodrähte mit einem Durch-
messer von 30-40 nm und einer Länge von
10-20 µm zu erhalten, wie festgelegt, unter Ver-
wendung von Rasterelektronenmikroskopie.

2. Herstellungsverfahren nach Anspruch 1, wobei die
Reaktionszeit im Reaktionskessel 9-10 Stunden be-
trägt.

Revendications

1. Procédé de préparation de nanofils d'argent avec un
rapport d'aspect uniforme comprenant les étapes
suivantes consistant à :

(1) dissoudre 0,16 g de nitrate d'argent à tem-
pérature ambiante dans 20 mL de glycérol pour
obtenir une solution A ;

(2) dissoudre 5 g de polyvinylpyrrolidone à tem-
pérature ambiante dans 80 mL de glycérol pour
obtenir une solution B ;

(3) mélanger de manière uniforme la solution A
et la solution B afin de former une solution C ; et

(4) ajouter 5 mL d'eau ultra pure dans la solution
C et mélanger de manière uniforme afin de for-
mer une solution D, finalement, transférer la so-
lution D dans une bouilloire à réaction, mettre la
bouilloire à réaction à une température définie

de 160 °C, et mettre fin à la réaction après un
certain temps de réaction ; diluer une solution
mère de nanofils d'argent dans la bouilloire à
réaction avec de l'alcool et centrifuger deux fois
pour obtenir des nanofils d'argent précipités
ayant un diamètre compris entre 30 et 40 nm et
une longueur comprise entre 10 et 20 µm, telle
que déterminée en utilisant la microscopie élec-
tronique à balayage.

2. Procédé de préparation selon la revendication 1,
dans lequel le temps de réaction dans la bouilloire
à réaction est compris entre 9 et 10 heures.

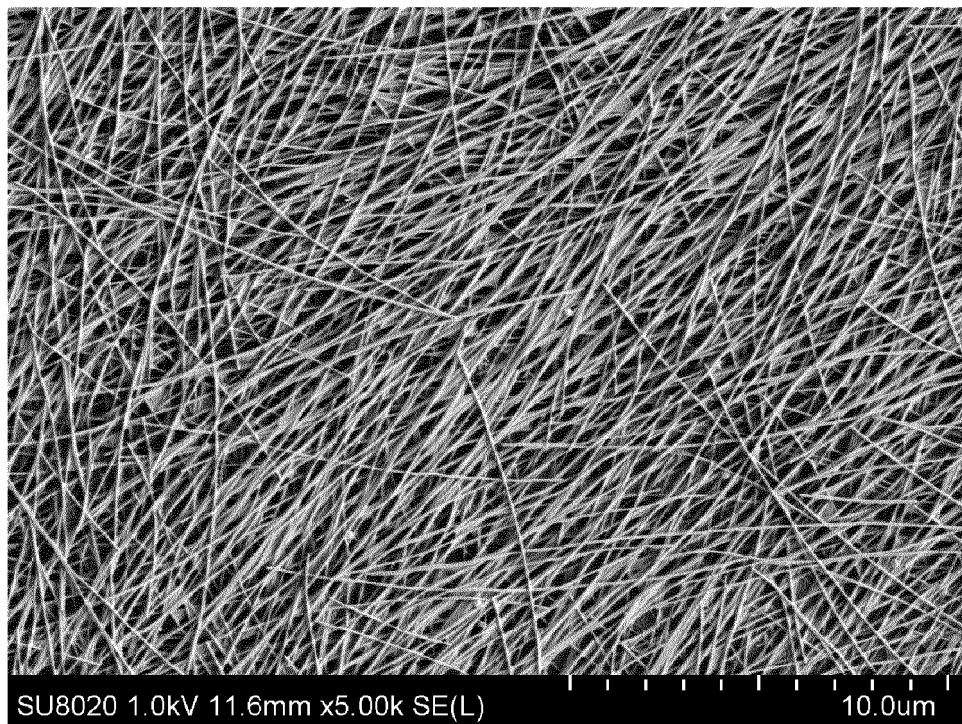


FIG. 1

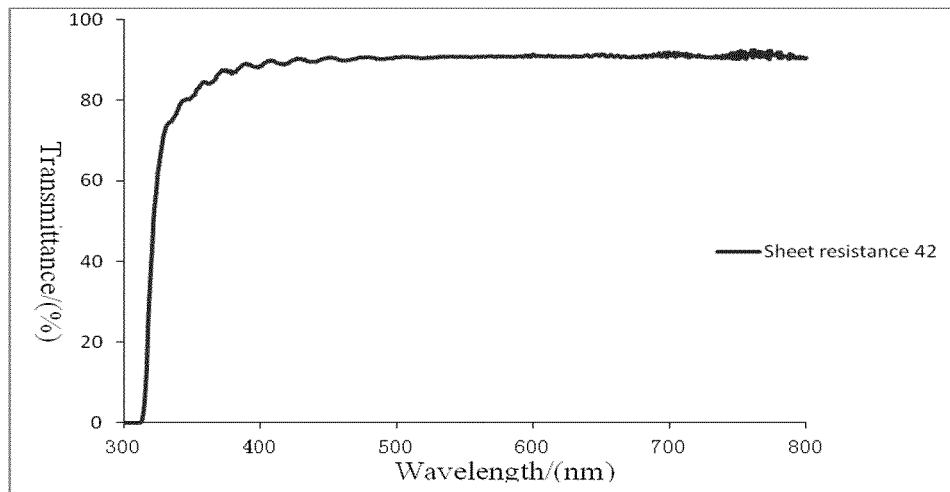


FIG. 2

REFERENCES CITED IN THE DESCRIPTION

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