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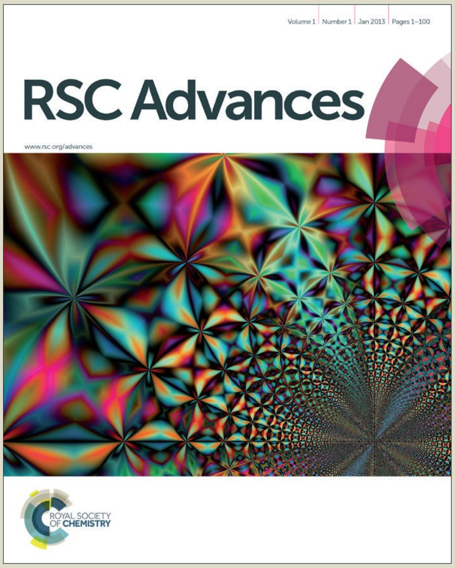

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Large Scale Preparation of Silver Nanowires with Different Diameters by a One-pot Method and Their Application in Transparent Conducting Films

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Silver nanowires (AgNWs) with varied diameters were synthesized by a facile and efficient one-pot polyol method. The effects of reaction conditions on the morphology of the as-prepared AgNWs as well as the transmittance and optoelectronic performance of the transparent conducting films made of the AgNWs were investigated. Findings indicate that each run of the reaction of the established synthesis protocol can provide more than 10 g of AgNWs, and the dynamic balance between the reduction of Ag^+ (yielding Ag^0) and the formation of AgCl nanocubes has an important effect on the yield and morphology of AgNWs. Based on the proper adjustment of the reaction conditions, the average diameter of AgNWs can be adjusted in the range of 40-110 nm while the yield of one-dimensional nanostructures in the products is as much as 95%. Furthermore, the as-synthesized AgNWs can be easily spin-coated onto polyethylene glycol terephthalate substrate to afford transparent conducting films with a transmittance of as high as 87% and a sheet resistivity below 70 Ω/sq, which demonstrates that the present synthesis strategy could be of special significance for the commercial preparation and application of silver nanowires as an alternate of commercial indium tin oxide film.

Introduction

Silver nanowires (denoted as AgNWs) have drawn much interest in recent years, due to their unique size-dependent optical,[1, 2] electrical,[3] and thermal properties.[4, 5] AgNWs are currently applied in nanoscale electronic devices such as surface-enhanced Raman scattering device[6] and biological sensors,[7, 8] and in particular, they can be used as an alternative of indium-tin oxide (denoted as ITO) to prepare transparent conducting films.[9, 10]

Thanks to its low sheet resistance (<100 Ω/sq) and high transmittance (~90%), ITO is the most widely used material of transparent conducting film. However, ITO is mechanically brittle and ITO film usually grown by a sputtering process is not suitable for flexible application.[11, 12] This drawback, fortunately, could be overcome by applying Ag nanowire film, since Ag nanowire film exhibits comparable or even better mechanical and electronic performances than ITO film.[13-15] Moreover, the transmittance and resistance of AgNWs are highly dependent on their diameter, which means it is imperative to develop a facile and efficient method so as to realize large scale production of AgNWs with controllable diameters. Currently available methods for producing AgNWs include template-guided synthesis,[16] solvothermal method[17] and electrochemical method.[18] Among these methods, polyol process with low cost, high yield and simple process is considered to be competitive. Xia et al. initially prepared AgNWs by polyol process in the presence of seeds.[19] Enlightened by Xia and co-workers´ research, many other researchers have made numerous efforts to improve the polyol synthesis in order to keep pace with the rapidly growing demand for AgNWs in nanotechnology.[20, 21]

Many researchers have investigated the effects of various reaction parameters (temperature, $AgNO_3$ concentration, molecular weight and concentration of polyvinylpyrrolidone (denoted as PVP), type and amount of salt, injection rate, and stirring rate) on the poyol synthesis of AgNWs.[21-25] These researches, unfortunately, are usually hard to be repeated, due to the difficulty to simultaneously control the ratios of reactants and the reaction temperature. Bernardino Ruiz et al. prepared ultralong silver nanowires using a one-pot polyol-mediated synthetic procedure.[26] However, their synthesis strategy relies on drop of one solution to another at a certain speed, which means it is infeasible to be enlarged in industrial preparation. Furthermore, few are currently available about the mutual influences of various reaction parameters, and it still remains a challenge to realize the mass production of silver nanowires.[27]

Bearing those perspectives in mind, in the present research we intend to develop a simple one-pot method to achieve high yield (>95%) and large-scale (over 10 g per pot) production of AgNWs with controllable diameters. Moreover, we adopt as-synthesized AgNWs as an alternate of ITO to prepare transparent conducting film *via* a simple spin coating method, and we investigate the transmittance and electronic performance of the as-fabricated transparent conducting film on polyethylene glycol terephthalate (denoted as PET) substrate. This article reports the effects of various reaction parameters on the morphology of AgNWs as well as the transmittance and sheet resistivity of AgNWs transparent conducting film.

Experimental

Materials

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$AgNO_3$, ethylene glycol (EG), and $NiCl_2·6H_2O$ were purchased from Sinopharm Chemical Reagent Company (Beijing, China). PVP (number averaged molecular weight: 55000) was supplied by SigmaAldrich. All the chemicals are of analytical purity and were used as received.

Preparation of AgNWs

Generally, EG acts as both solvent and reducing agent. PVP serves as the capping agent, chloride salts serves as the control agent, and $AgNO_3$ serves as the precursor in traditional polyol process.[19] Briefly, a 5-L reaction kettle was performed to directly dissolve 30 g of $AgNO_3$, 60 g of PVP and 25 mL of 0.03 M $NiCl_2$/EG solution in 3.6 L of EG (relevant reaction details are listed in Table 1). Resultant mixed solution was electrically heated to 100 ^{o}C and held there for 30 min, thereby allowing the solution to become dark red. Then the solution was heated to 140 ^{o}C within 20 min, thereby allowing the reaction solution to turn into light gray green in a period of time. Upon completion of heating, the reaction solution was cooled to room temperature naturally. Resultant suspension was diluted with water (at a volume ratio of 1 : 2) and centrifuged twice at 4000 rev/min (each for a duration of 5 min) in order to separate excess PVP from the AgNWs. As-obtained AgNWs products were dispersed in ethanol and stored at room temperature for further characterization.

Fabrication of transparent electrodes

PET substrates were soaked in hydrogen peroxide solution for 2 h in order to improve hydrophilicity. The soaked PET substrates were then sequentially cleaned with water and ethanol. Onto the as-cleaned PET substrates was finally spin coated the dispersion solution of the as-obtained AgNWs with different diameters at a rotary rate of 3000 rev/min rate for 60 s, with which the performance of the as-coated transparent conducting films was accommodated through adjusting the rotary rate and coating time.

Characterization

Scanning electron microscopic (SEM) images were obtained with a Nova NanoSEM450 instrument. The transmission electron microscopic (TEM) images were obtained with a JEOL JEM-2010 instrument. A drop of the suspension of the as-prepared AgNWs in ethanol was dipped onto the copper grids coated with amorphous carbon film, thereby affording the sample for TEM analysis. Powder X-ray diffraction (XRD) patterns were recorded with a D8-ADVANCE diffractometer (Cu Kα radiation; 40 kV, 60 mA). The sheet resistance of the electrodes was measured with a four-probe surface resistivity meter (ST2258C; Jingge Company, China). Transmittance spectra were recorded with a PE Lambda 950 device (the substrate was used as a reference).

Table 1 Reaction conditions and size of as-prepared AgNWs

Sample	$AgNO_3$ (M)	PVP (M)	$NiCl_2$ (mM)	Temperature 1 (^{o}C)	Temperature 2 (^{o}C)	Reaction time (min)	Diameter (nm)	Length (μm)
a1	0.05	0.3	0.4		100	240		
b1	0.05	0.3	0.4	100	120	150	44	15
c1	0.05	0.3	0.4	100	140	90	47	35
d1	0.05	0.3	0.4	100	160	60	55	20
e1	0.05	0.3	0.4	100	180	40	65	3
f1	0.05	0.3	0.4	100	200	20		
a2	0.025	0.3	0.4	100	140	90	45	8
b2	0.05	0.3	0.4	100	140	90	47	35
c2	0.1	0.3	0.4	100	140	90	54	15
d2	0.15	0.3	0.4	100	140	90	75	10
a3	0.05	0.075	0.4	100	140	90	100	25
b3	0.05	0.15	0.4	100	140	90	42	28
c3	0.05	0.3	0.4	100	140	90	47	35

d3	0.05	0.45	0.4	100	140	90	53	25
e3	0.05	0.6	0.4	100	140	90		
a4	0.05	0.3	2	100	140	90		
b4	0.05	0.3	1	100	140	90	90	5
c4	0.05	0.3	0.4	100	140	90	73	10
d4	0.05	0.3	0.2	100	140	90	44	35
e4	0.05	0.3	0.1	100	140	90	47	35
f4	0.05	0.3	0.025	100	140	90		
a5	0.05	0.3	0.025	100	160	90	65	10
b5	0.05	0.3	0.025	100	180	90	90	5
c5	0.05	0.3	0.025	100	200	90	100	3
d5	0.075	0.15	0.05	100	160	90	110	20

Results and discussion

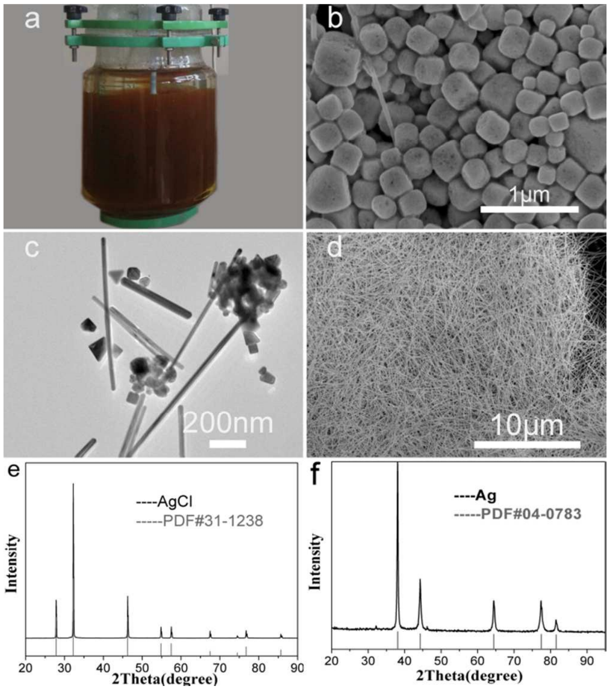

Fig. 1 (a) Optical photograph of reaction solution in 5-L kettle at 100 °C; (b) SEM image of AgCl nanocubes prepared at 100 °C; (c) TEM image of MTPs growing into AgNWs; (d) SEM image of AgNWs prepared by one-pot method; (e) XRD pattern of AgCl nanocubes; (f) XRD pattern of AgNWs.

Characterization of AgNWs

The one-pot synthesis of AgNWs involves three stage of reactions. The first stage is the formation of AgCl nanocubes as heterogeneous nuclei at 100 °C (Fig. 1b and e) in association with the solution colour change into dark red (Fig. 1a).[28] The second stage is the formation of the multiply twinned particles (MTPs; Fig. 1c) which would separate from the nanocubes and grow into AgNWs.[29] The third stage refers to the growth of AgNWs under specific conditions along with the formation of Ag nanoparticles. Fig. 1d shows the typical SEM images of the as-prepared AgNWs. It can be seen that the as-prepared Ag nanowires have a mean diameter of 45 nm, a length of 40 μm, and an aspect ratios of about 1000. Besides, the yield of the one-dimensional (1D) nanostructures is estimated to be over 95%. X-ray diffraction (XRD) pattern in Fig. 1f shows that the as-synthesized AgNWs can be indexed to face-centered cubic phase Ag belonging to the space group Fm3m (225) (JCPDS file No. 04-0783).

Effect of temperature on the morphology of AgNWs

Xia et al. found that heating ethylene glycol (EG) in air results in its oxidation to glycolaldehyde (GA), which has a profound impact on the nucleation and growth kinetics of AgNWs and the reducing power of EG is dependent on temperature.[30] Wiley et al. reported that the Ag nanowires grew longer and wider at lower temperatures as the nucleation rate was lower at lower reaction temperatures, leaving more silver precursor per nuclei.[31]

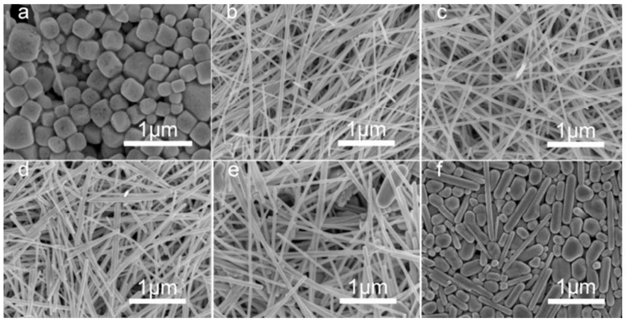

Fig. 2 SEM images of Ag nanowires synthesized at different temperature: (a) 100 °C, (b) 120 °C, (c) 140 °C, (d) 160 °C, (e) 180 °C, and (f) 200 °C.

Generally, when the reaction temperature is below 120 °C, AgNWs cannot be obtained. Thus the first stage reaction temperature is selected as 100 °C to prepare AgCl nanocubes. Fig. 2 shows the SEM images of AgNWs obtained at different temperature. It can be seen that only AgCl nanocubes are obtained when the reaction temperature is 100 °C (Fig. 2a). As temperature rises (corresponding to items a1-f1 in table 1), AgNWs with different diameters are obtained, and their diameter tends to rise with elevating temperature while their length tends to decrease in association with the increase of the amount of nanoparticle byproducts (Fig. 2(b-e)). When the temperature is as high as 200 °C, only silver nanoparticles and nanorods are obtained (Fig. 2f). Besides, the reaction process tends to be increasingly accelerated as the temperature increases, which is because the reaction temperature decides the reduction rate of Ag^+ (yielding Ag^0). When the reaction temperature is too high (the reaction rate is too fast), Ag atoms are deposited not only on {100} but also on {111}, while the total amount of Ag atoms is constant. As a result, the as-prepared AgNWs become wider and shorter as the temperature increases. In the meantime, elevating temperature facilitates the accelerated generation of Ag atoms, thereby allowing more Ag atoms to form Ag nanoparticles rather than to be deposited on MTPs.

Effects of $AgNO_3$ concentration on morphology of AgNWs

$AgNO_3$ acts as the precursor, and its concentration is vital to the yield and morphology of AgNWs, because it can expand the preparation scale and change the aspect of AgNWs.[27, 32] The SEM images of the AgNWs synthesized at different $AgNO_3$ concentrations (corresponding to items a2-d2 in table 1; the other parameters in the typical polyol process are kept constant) are shown in Fig. 3 (a-d). It can be seen that AgNWs tend to become wider and shorter as the concentration of $AgNO_3$ increases, which is consistent with what is reported elsewhere.[27] Besides, when the $AgNO_3$ concentration is too high or too low, the amount of nanoparticles increases substantially, as evidenced from corresponding SEM images (Fig. 3).

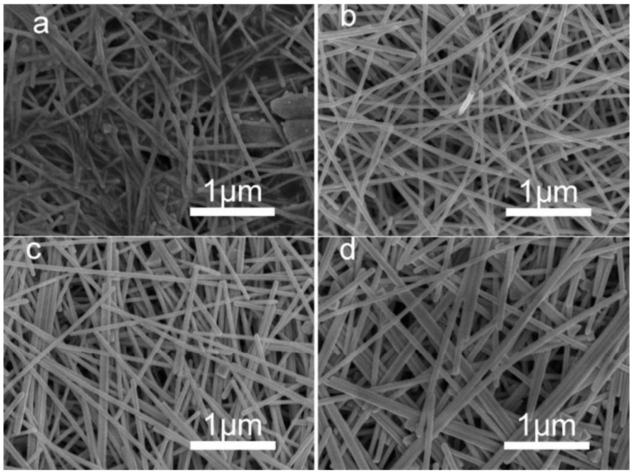

Fig. 3 SEM images of silver nanowires synthesized at different $AgNO_3$ concentration: (a) 0.025 M, (b) 0.050 M, (c) 0.100 M, and (d) 0.150 M.

Particularly, as the $AgNO_3$ concentration is 0.05 M, the least byproducts are produced. Thus we select 0.05 M as the optimal concentration of $AgNO_3$.

Effects of PVP/$AgNO_3$ molar ratio on morphology of AgNWs

Xia et al. proved that the anisotropic growth of AgNWs is maintained by selectively covering the facets with PVP while leaving the facets largely uncovered by PVP and thus highly reactive.[33] The final morphologies of Ag nanostructures at the end of the polyol process are strongly dependent on the PVP/$AgNO_3$ molar ratio [34] and the weight of PVP chain.[35, 36] The effect of PVP/$AgNO_3$ molar ratio on the morphology of AgNWs is shown in Fig. 4 (a-d) (corresponding to items a3-e3 in table 1). When the PVP/$AgNO_3$ molar ratio is too small, the as-prepared AgNWs have non-uniform morphology, and a large amount of byproducts is obtained, since the {100} planes cannot be adequately passivated. When the PVP/$AgNO_3$ molar ratio is too large, no AgNWs are obtained,

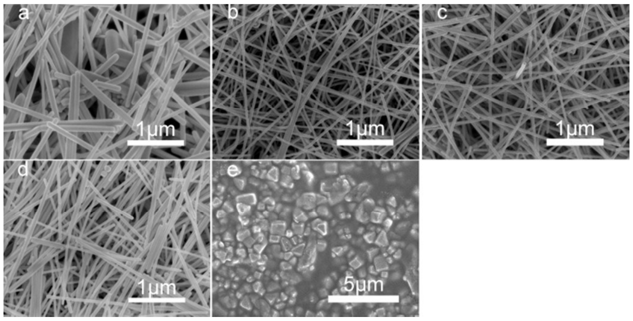

Fig. 4 SEM images of silver nanowires synthesized at different

PVP/$AgNO_3$ molar ratio: (a) 1.5:1, (b) 3:1, (c) 6:1, (d) 9:1, and (e) 12:1.

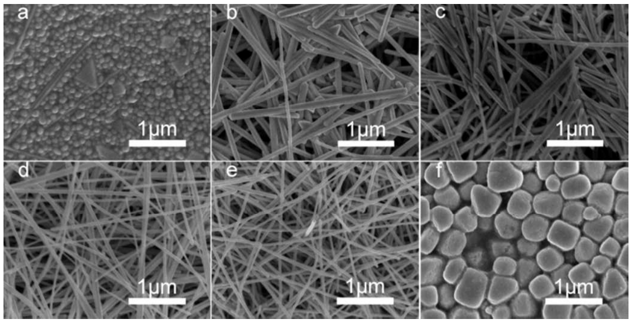

Fig. 5 SEM images of silver nanowires synthesized at different $NiCl_2$/$AgNO_3$ molar ratio: (a) 1:2000, (b) 1:1000, (c) 1:500, (d) 1:250, (e) 1:100, and (f) 1:25.

because in this case the {111} planes are completely passivated (Fig. 4e). Therefore, in order to obtain AgNWs with the least byproducts and uniform morphology, we select the optimal $AgNO_3$/PVP molar ratio as 1:6.

Effect of $NiCl_2$/$AgNO_3$ molar ratio on morphology of AgNWs

NaCl and $CuCl_2$ or $FeCl_3$ are often used as the control agents for synthesizing AgNWs by polyol process.[38-40] It has been found that the control agents play an important role in the formation of AgCl nanocubes and the production of silver nanowires, since AgCl nanocubes induce the heterogeneous nucleation of metallic Ag.[28] Chou et al. obtained AgNWs with controllable diameters by changing the concentration of chloride ions.[37] In the present research, we select $NiCl_2$ as the control agent. Fig. 5 (a-f) show the SEM images of the AgNWs products prepared at $NiCl_2$/$AgNO_3$ molar ratio from 1:2000 to 1:25 (corresponding to items a4-f4 in table 1). It can be seen that no AgNWs are obtained when the amount of $NiCl_2$ is too little, which is because few AgCl nanocubes are formed in this case (Fig. 5a). When the $NiCl_2$/$AgNO_3$ molar ratio is too high, no AgNWs are obtained, because in this case no enough free Ag atoms are deposited and grow into nanowires (Fig. 5f). With the decrease of $NiCl_2$/$AgNO_3$ molar ratio, the diameter of AgNWs tends to decrease while their length tends to increase. Moreover, the amount of nanoparticles tends to increase when the dosage of $NiCl_2$ decreases, and the highest yield is achieved at a $NiCl_2$/$AgNO_3$ molar ratio of 1:100.

Interaction between different reaction parameters

Buhro et al. demonstrated that the AgCl nanocubes are decorated by Ag nanoparticles, and some of them are pentagonally twinned and grow into nanowires.[29] They also found that most of the AgCl nanocubes are degraded during the course of the growth process, releasing Ag nanoparticles and nanowires which are separated during the workup. These studies remind us that increasing $AgNO_3$ concentration and temperature or decreasing $NiCl_2$ concentration will lead to more silver nanoparticle byproducts during the synthesis of AgNWs with large diameters. In the present research, we have found that few AgNWs are obtained at a $NiCl_2$/$AgNO_3$ molar ratio of 1:2000 and a temperature of 140 °C (Fig. 5a) or at a $NiCl_2$/$AgNO_3$ of 1:100 and a temperature of 200 °C (Fig. 2f). However, when $NiCl_2$/$AgNO_3$ molar ratio is kept as 1:2000, AgNWs can be obtained at 160 °C, 180 °C, and even 200 °C (Fig. 6(a-c)) (corresponding to items a5-c5 in table 1) in association with distinct decrease of the amount of silver nanoparticles.

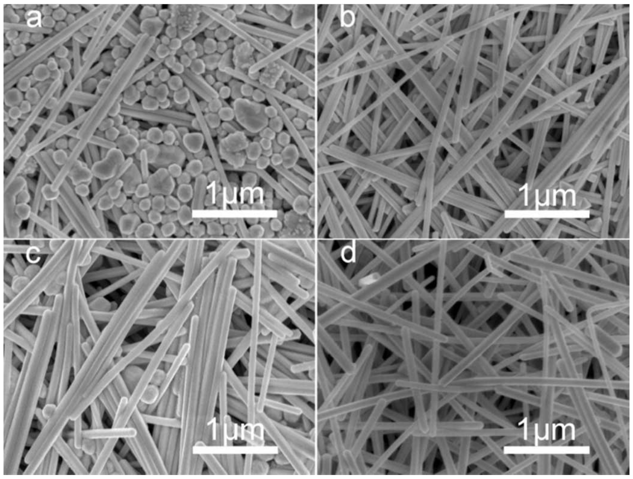

Fig. 6 SEM images of silver nanowires synthesized at different temperature: (a) 160 °C, (b) 180 °C, and (c) 200 °C (the $NiCl_2$/$AgNO_3$ molar ratio is 1:2000) and SEM image of AgNWs nanowires with uniform morphology produced at 160 °C, 0.05 M $AgNO_3$, $AgNO_3$/PVP molar ratio of 1:6, and $NiCl_2$/$AgNO_3$ molar ratio of 1:1000 (d).

It is known that temperature decides the reduction rate of Ag^+ (yielding Ag^0) and $NiCl_2$ concentration decides the amount of AgCl nanocubes. Therefore, it can be speculated that with the formation of MTPs and their separation from AgCl nanocubes, new Ag atoms are attached to AgCl nanocubes heterogeneous nuclei. The reaction mechanism for synthesizing AgNWs is shown in Fig. 7. When the reduction rate of Ag^+ is too fast or the amount of $NiCl_2$ is too small, too much free Ag atoms are attached to AgCl nanocubes and grow into nanoparticles directly. Therefore, if the reduction of Ag^+ and the formation of AgCl nanocubes could reach a dynamic balance, the amount of nanoparticle byproducts would be efficiently decreased. Based on this assumption, we properly adjusted all reaction parameters so as to prepare wide AgNWs with few nanoparticles (see item d5 in table 1). As it can be seen in Fig. 6d, corresponding AgNWs have uniform morphology (average diameter: 100 nm; yield: >85%).

Transparent electrodes

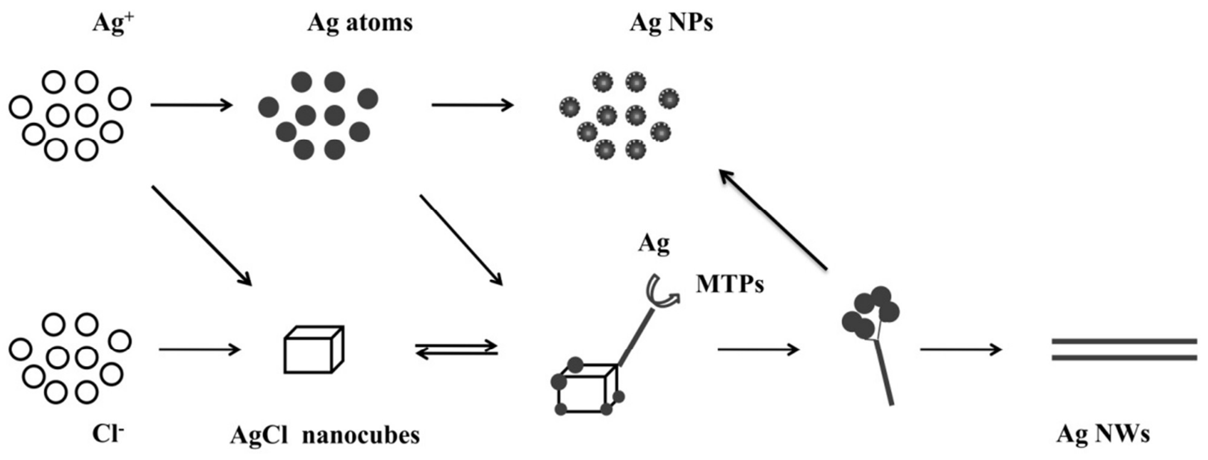

Fig. 7 The mechanism of AgNWs' Nucleation and growth process.

The performance of AgNWs-based transparent conducting films tends to be improved with decreasing diameter and increasing AgNWs aspect ratio, since thin AgNWs scatter less light and increasing aspect ratio decreases the number of high-resistance nanowire-nanowire contacts in the film.[31, 41] To fabricate transparent conducting films with different optoelectronic performance, we spin-coated the dispersion solution of the as-prepared AgNWs with different diameters onto the pre-processed PET substrates by adjusting the rotary speed and the time of spin coating process. As shown in Fig. 8(a, c, e), the resistance of the conducting films increases with the increase of the transmittance; and the photoelectric properties of the conducting films tend to be increasingly worsened as the diameter increases. Moreover, as shown in Fig. 8(b, d, f), the dispersion of AgNWs on the PET substrate tends to be increasingly improved as the diameter of AgNWs declines, which is consistent with previous studies. The transmittance of the as-fabricated AgNWs-based transparent conducting film is above 87%, and its square resistance is below 70 Ω/sq, which indicates that the optoelectronic performance of the AgNWs-based transparent conducting film is comparable to the performance of ITO.

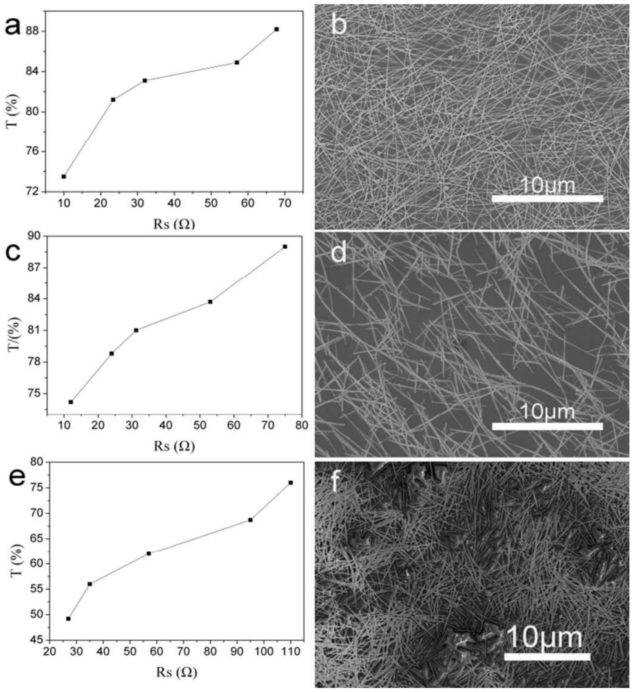

Fig. 8. (a, c, e): Transmittance and resistance of transparent conducting films prepared from AgNWs with different diameters (45 nm, 70 nm, 100 nm). (b, d, f): SEM images of the conducting films prepared from AgNWs with different diameters (45 nm, 70 nm, 100 nm).

Conclusion

A simple and efficient polyol-based one-pot method has been established to realize large-scale synthesis of silver nanowires. The method can be applied to fabricate AgNWs with controllable diameters in a high yield while the dosage of the reactants and the reaction temperature are properly adjusted. Besides, the as-prepared AgNWs can be adopted to fabricate transparent film electrodes with a transmittance above 87% and a square resistance below 70 Ω/sq, which means that the present method might be promising for the commercial preparation and application of silver nanowires.

Acknowledgements

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Silver nanowires (AgNWs) with varied diameters were synthesized by a facile and efficient one-pot polyol method. Each run of the reaction with this method can provide more than 10 g of AgNWs.