

Preparation of Silver Nanowires by Polyol Method and Comparison of Transparent Conductive Films of Silver Nanowires Coated on PET and Glass Substrate

NAY CHYI HTWE, GAN GUOYOU

(Material Science Department, Kunming University of Science and Technology, Kunming 650093, China)

Abstract: Different ratios of poly(vinylpyrrolidone) polymer (PVP) and AgNO_3 were used for the production of silver nanowires (AgNWs) by using polyol method^[1]. PVP was used as the stabilizer and ethylene glycol (EG) was used as the reducing reagent. There are four types of samples which were prepared by using AgNO_3 /PVP with different ratios: the sample D of AgNWs has a more uniform diameter and less impurity than the others. AgNWs lengths and diameter were controlled by changing the AgNO_3 /PVP ratio, injection rates and different molecular weight of PVP. We demonstrate that the preparation of AgNWs with a thin diameter (17~100 nm) and long length (30 nm to above 100 μm) were used by polyol method. AgNWs' structures were characterized by XRD, SEM, TEM and UV-vis, respectively. The fabrication of transparent conductive films of poly ethylene terephthalate(PET) and glass substrate using a scalable lamination-assisted solution method and spin-coating process is described in this study. For transparent conductive film of PET and glass substrate, AgNWs inks with a resistance of 17, 26 Ω/sq and transmittance of 88%, 80% respectively were produced using a mixture of AgNWs with high and low aspect ratio.

Key words: poly(vinylpyrrolidone) polymer(PVP); silver nanowires(AgNWs); AgNO_3 /PVP; transparent conductive films; poly(ethylene terephthalate) PET; glass substrate

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银纳米线的聚醇法制备及其在聚酯和玻璃基板上镀成的透明导电薄膜对比

NAY Chyi Htwe, 甘国友

(昆明理工大学 材料学院, 昆明 650093)

摘 要: 以聚乙烯基吡咯烷酮(PVP)为稳定剂, 聚乙二醇(EG)为还原剂, 采用聚醇法^[1]制备了银纳米线。根据硝酸银/PVP 比例的不同制备了 4 种类型的银纳米线样品, 其中样品 D 直径最均匀, 杂质含量最低。通过改变硝酸银/PVP 比例、加入速率和 PVP 分子量, 可对银纳米线的长度和直径进行控制, 得到了小直径(17~100 nm)、大长度(30 nm 到 100 μm 以上)的银纳米线。采用 XRD、SEM、TEM 和紫外-可见光谱对银纳米线的结构进行了表征。采用不同径长比纳米银线混合, 以聚对苯二甲酸二乙酯(PET)和玻璃为衬底, 分别使用可扩展层压辅助溶液法和旋转涂布法制备了 2 种透明导电薄膜, 其方阻分别为 17 和 26 Ω/sq , 透光率分别为 88%和 80%。

关键词: 聚乙烯基吡咯烷酮(PVP); 银纳米线(AgNWs); AgNO_3 /PVP; 透明导电薄膜; 聚对苯二甲酸二乙酯(PET); 玻璃基板

Silver nanowires (AgNWs) have the diameter in the range of 10^{-9} meters, its lengths are between 10^{-9} and 10^{-6} meters and also have fascinating optical, electrical, catalytic, thermal and other related properties. AgNWs can also be defined as that width ratio to length ratio are being smaller than 1000 nm. Additionally, The structure of nanowires that have a thickness, diameter is constrained to micrometers or nanometers and an unconstrained length. The electrical conductivity (6.3×10^7 S/m) of silvers are higher than the other metals. AgNWs may be used as the most important candidates for the transparent and flexible electronics^[2]. Nano wires are used in many application like electronic device, nano electronic device, sensor, LED, nanoscale quantum devices, additives composites electrode and solar cell. The top-down and bottom-up methods are used for the synthesizing of AgNWs. A top-down method was used for the reducing of a large fragments material to small fragments materials and also used like lithography or electrophoresis. The nanowires in which combining constituent adatoms were synthesized by using the bottom-up method. Nanowire thermal treatment step are also used and a formation of self-limiting oxidation are often involved, to the formation of the controlled aspect ratio and size of the structures^[3]. In addition, AgNWs preparation was needed to much more attention because silver has the higher thermal and electrical conductivity than the other metals and silver are also used in many applications that the processing of silver into one-dimensional (1D) nanostructure was performed to enhance it^[4]. The AgNWs with longer length and smaller diameter can perform a good electrical active network by providing shorter contact resistance between nanowire junctions. Hence, the longer length AgNWs with smaller diameter are good for better performance using a simple solution process method, and it is of great interest and challenge in the transparent and flexible electronic industry^[5]. Many researcher were interesting in the synthesis of nanowires because it is widely used in many electronic. AgNWs have been synthesized by many method like polyol process, hydrothermal method, wet chemical

synthesis, and ultraviolet irradiation photo reduction techniques. In addition, many researcher were more used the polyol process than the other method because it is low coast, effective, high yield and simple process^[6]. The polyol process in which an exotic reagent leads to the formation of wire like structure. In this report, we were prepare the AgNWs with long length and thin diameter by using the polyol method with different ratio of AgNO_3/PVP , different injection rates and different molecular weight of PVP. Extensive work has been performed to develop the synthesis of nanowires made of silver, as it exhibits the lowest resistivity at room temperature out of all the elements. This effort led to the discovery of a method to fabricate nanowires with very large aspect ratios (length divided by the diameter). Their low resistivity and high aspect ratios allow the fabrication of networks which can simultaneously conduct electricity and be transparent to visible light. These are the two most essential properties required for a transparent electrode. The goal of this thesis is to explore the fabrication of randomly-oriented silver nanowire networks as an alternative transparent conductive material for use in solar cells or display technologies. In particular we describe the motivation for alternative transparent conductive materials to replace Indium Tin Oxide, and propound the benefits of silver nanowire networks. Silver nanowire networks have already been shown to produce similar electro-optical properties as commercial ITO^[7-9].

1 Experimental

1.1 Materials

The following materials were purchased commercially and were used without further purification. Silver nitrate (AgNO_3 , Aladdin, 99.8%), poly(vinyl)pyrrolidone (PVP, M_w : 24000, 58000, 360000 and 1300000 g/mol, Sigma-Aldrich), and copper(II) chloride (CuCl_2 anhydrous, 99.0%, Aladdin) were used for preparation of AgNWs. Ethylene glycol (EG, 99.0%, Aladdin) was used both as a solvent and reducing agent. Ethyl alcohol (99.5%, Feng Chuan Chemicals) and acetone (99.8% KESHI Chemicals)

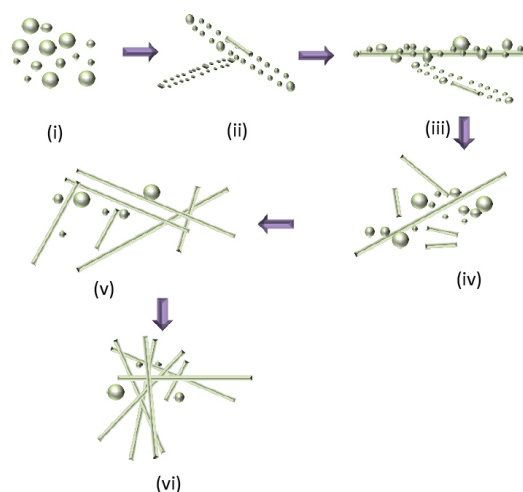
were used as washing solvents. De-ionized water was used for re-dispersion of AgNWs. Isopropyl alcohol (99.5%, Aladdin) was used for suspension of AgNWs and HCl (99.0%, Aladdin) was also used for remove oxidizing of silver nanowire films.

1.2 Preparation of AgNWs

We synthesized AgNWs by using polyol method have been reported previously with some adjustments^[10]. Fig.1 shows the schematic growth of AgNWs and nanostructures coarsening mechanism. A three-neck flask was filled with 100 mL of EG and constant heated at 170°C for 45 min. And then a PVP solution (0.7mol/L in 10 mL of EG solution) was added to the flask. After 3 min, 1.15 mL of copper(II) chloride solution (4mmol/L, 4.6×10^{-6} mol in EG solution) poured into the flask. Copper(II) chloride solution has been used to etch silver atoms to form multi-twinned structural particles for the growth of wires. After 30 min, silver nitrate solution 1.08 g (0.7 mol/L), 0.54 g (0.35 mol/L), 0.27 g (0.17 mol/L) and 0.135 g (0.08 mol/L), 10 mL in EG was injected into flask by different rates using with a 10 mL of syringe. The polyol synthesis process in which temperature controller and a magnetic mixer that must be employed for the maintaining of the constant temperature of 170°C and constant stirring rates and then after the 60 min the reaction was stopped and the solution was allowed to cool at the room temperature. The AgNO₃/PVP molar ratios were 1, 2, 4 and 8 respectively. Then the solution of separated products were centrifuged at 7000 r/min for 30 min and it was diluted with acetone, washed many time with ethanol until the clear solution was obtained. After washing, AgNWs were re-dispersed in DI water. And then the purification process was again and again until to get the purified products that were AgNWs. It was placed in the oven at 100°C for drying.

1.3 Preparation of silver ink for transparent conductive film

Isopropyl alcohol (IPA) was employed for the suspension of AgNWs (25 mg/mL). And then IPA was also used for the dilution of small amount of dispersion that it was down to the concentration 1 mg/mL. It was put in a sonic bath for 1 hr sonication.



Sample solution A to D: (i). 15 min; (ii). 25 min; (iii). 35 min; (iv). 45 min; (v). 55 min; (vi). 60 min

Fig.1 Schematic diagram of the growth of AgNWs and nanostructures coarsening mechanism^[11]

图 1 银纳米线生长和纳米晶粒粗化机理示意

Afterward, it was coated on 20×60 mm poly ethylene terephthalate (PET) substrates by passing through a laminator (GBC4500) at 5 mm/s. And then this suspension was also coated to a 10×30 mm glass substrates by spin coating process and then air dried. In addition, if necessary the Ag NW layers would be coated on the initial layer. Fig.8 shows the transparent films of PET and glass substrate. In this report, three ANWs samples as I($L=30\ \mu\text{m}$, $d_t=30\ \text{nm}$), II($L=50\ \mu\text{m}$, $d_t=60\ \text{nm}$), and III($L=70\ \mu\text{m}$, $d_t=100\ \text{nm}$) were used. The length and diameter of this samples I to III nanowires were arranged in the increase of ascending order. The AgNWs surface were easily oxidized and it's conductivity was decreased. So concentrated HCl was used to remove the oxidation on the AgNWs layer and then the film's conductivity were also increased. And then thin film of PET substrate and glass substrate were be incubated by using the volatilization of the concentrated HCl vapor at room temperature for 5~10 min. During the weak adhesion of Ag NW film coated on PET substrate and glass substrate were be easily scratched and damaged^[12].

1.4 Optical transmittance measurement and SEM

AgNWs images were taken by using the scanning electron microscopy (SEM) and field emission

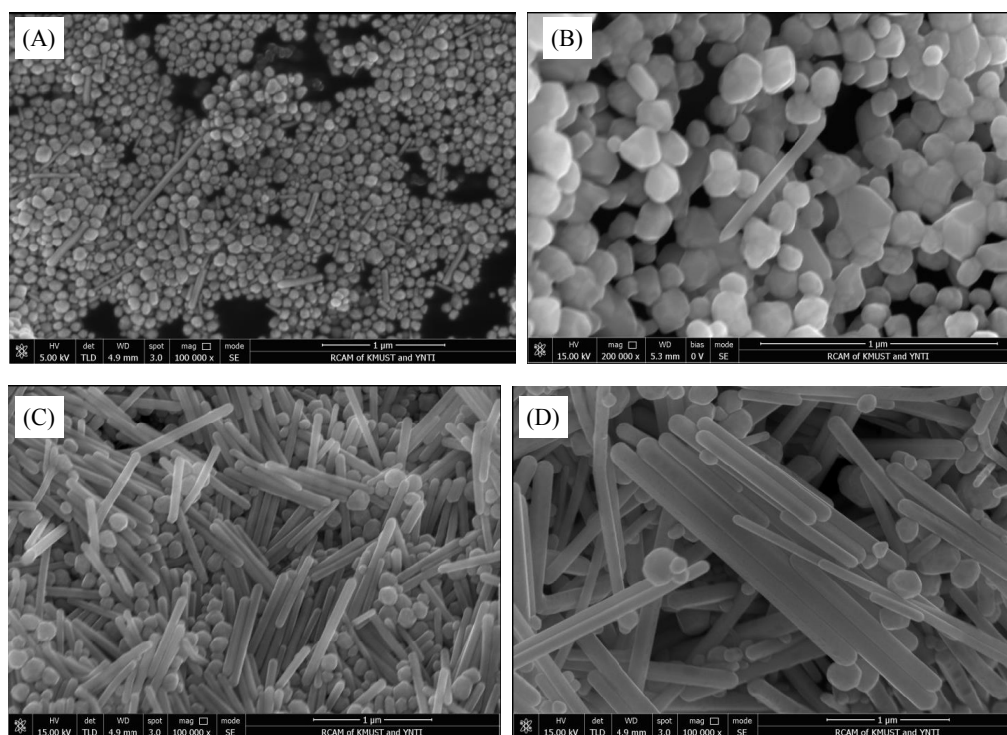
electron microscopy (FE-SEM). The structure of AgNWs were confirmed using the Siemens D 5000 X-ray diffractometer (Cu-K α radiation, nm) and the Libra 120 model transmission electron microscopy (TEM) using accelerating voltage of 400 kV. The evolution of AgNWs and transmittance of the conductive film were monitored by UV-vis spectroscopy (S-4100, Scinco). The conductivity of the transparent, conductive samples was measured

using a four-point probe (CMT- SR20000N) The resulting spectrum are shown in Fig.2, 3, 5, 6, 7 and 8.

2 Results and discussion

2.1 Preparation of AgNWs

Fig.2 shows the different ratio of AgNO₃/PVP is one of the most important parameter in the preparation of AgNWs via polyol process^[13].



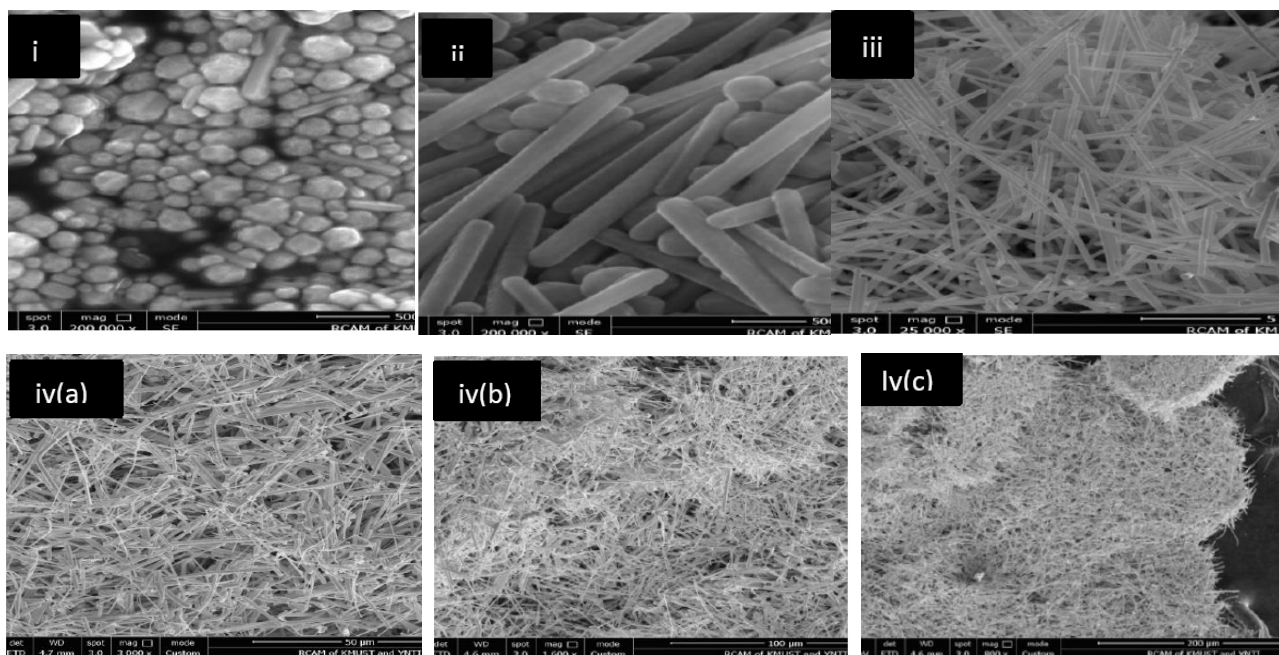
AgNO₃ in sample (A). 0.08; (B). 0.17; (C). 0.35; (D). 0.7 mol/L with 0.7 mol/L PVP respectively

Fig.2 SEM images of AgNWs in the presence of different silver nitrate/PVP molar ratio with constant rates (the diameters of the nanowires could be controlled from 17 to 100 nm)

图2 相同加入速率, 不同 AgNO₃/PVP 摩尔比条件下所得银纳米线(A~D)的 SEM 图像(直径可控制在 17~100 nm)

AgNWs were synthesized with different ratio of AgNO₃ and PVP (sample A to D) by polyol method. Sample D has more uniform diameter and less impurity than others. The SEM images of the AgNWs sample D with different injection rates is shown in Fig.3. The morphology of a nanowire like a cylinder is confirmed by using SEM images. SEM image of nanowires sample D cleaned by purification and centrifugation. Fig.4 exhibits the changes in diameter and length of AgNWs prepared with different injection rates at a reaction temperature of 170°C. As injection

rates of the silver source was increased from 1 to 7 mL/min. Monodispersed AgNWs of average diameter and length are plotted in Fig.4. The SEM images were analyzed by Image J software for the average diameter and length of AgNWs^[14]. Fig.5 shows the TEM images of the AgNWs (1 mL/min) and it indicate that a narrow size distribution and the uniform diameter of each AgNWs. Fig.5 shows that the structure of the AgNWs were be provided by using high resolution TEM images. The image was determined that the [110] direction of AgNWs were grown.



(i). 7 mL/min; (ii). 5 mL/min; (iii). 3 mL/min; (iv). 1 mL/min

Fig.3 SEM images of the effects of injection rate on the diameter and length of AgNWs sampled D

图 3 不同注入速率所得银纳米线样品 D 直径和长度的 SEM 图像

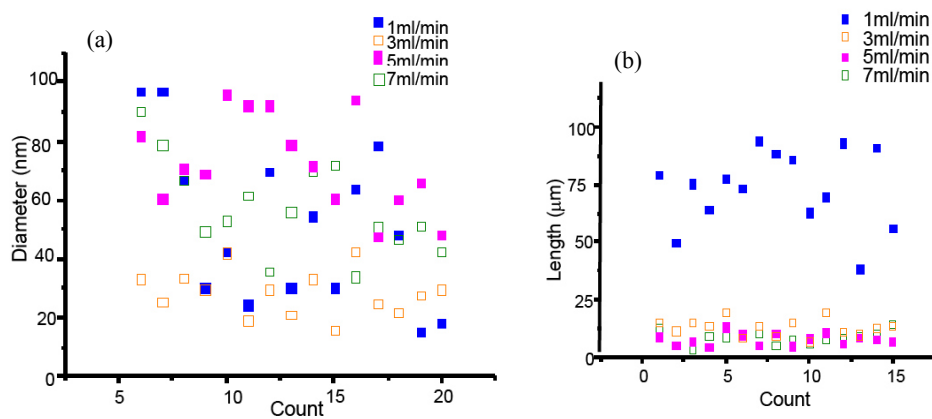


Fig.4 AgNWs of (a) diameter and (b) length with different injection rate

图 4 不同注入速率所得银纳米线的直径(a)和长度(b)

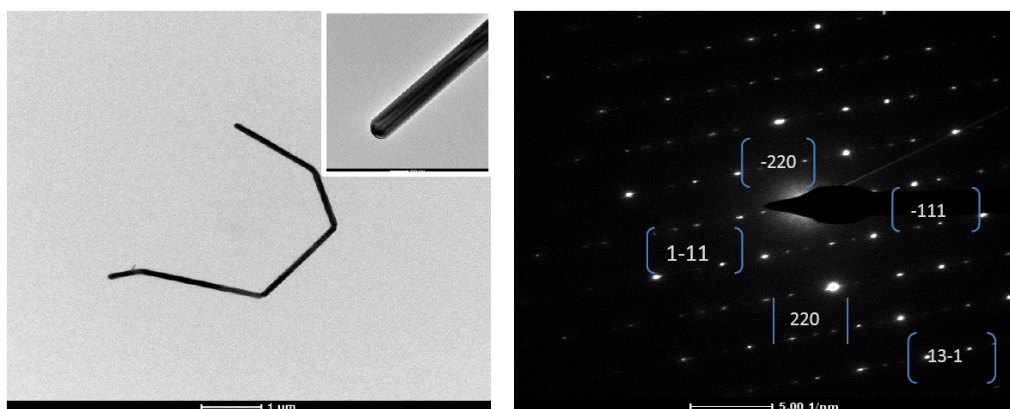


Fig.5 TEM images of AgNWs sample D with injection rate 1 mL/min produced by polyol

图 5 注入速率为 1 mL/min 所得银纳米线样品 D 的 TEM 图像

In addition, this Fig.5 also displays top of an Ag NW, and the contrasting was clearly confirmed that the Ag NW was equally separated by the longitudinal axis parallel to a twin plane. In recent work, a low threshold for the [111] face of Ag parallel to the twinning plane has demonstrated and which was also confirmed that in along the [111] plane, bi-crystals twinned have been grown.

In addition, extremely pure and uniform of AgNWs was capped with PVP and through the

synthesis, we were obtained that the less than 1 nm in thickness of Ag. Two dipole resonance peaks (355, 375 nm) of 1-D nanostructures (355 nm, 375 nm) shown in Fig.6(a). The shape of symmetry and the number of resonance peaks are affected on the intensity of peaks and polarization of the electron density of a nanoparticle^[19]. Fig.6(b) shows the AgNWs were synthesized by using polyol method comprised pure phase.

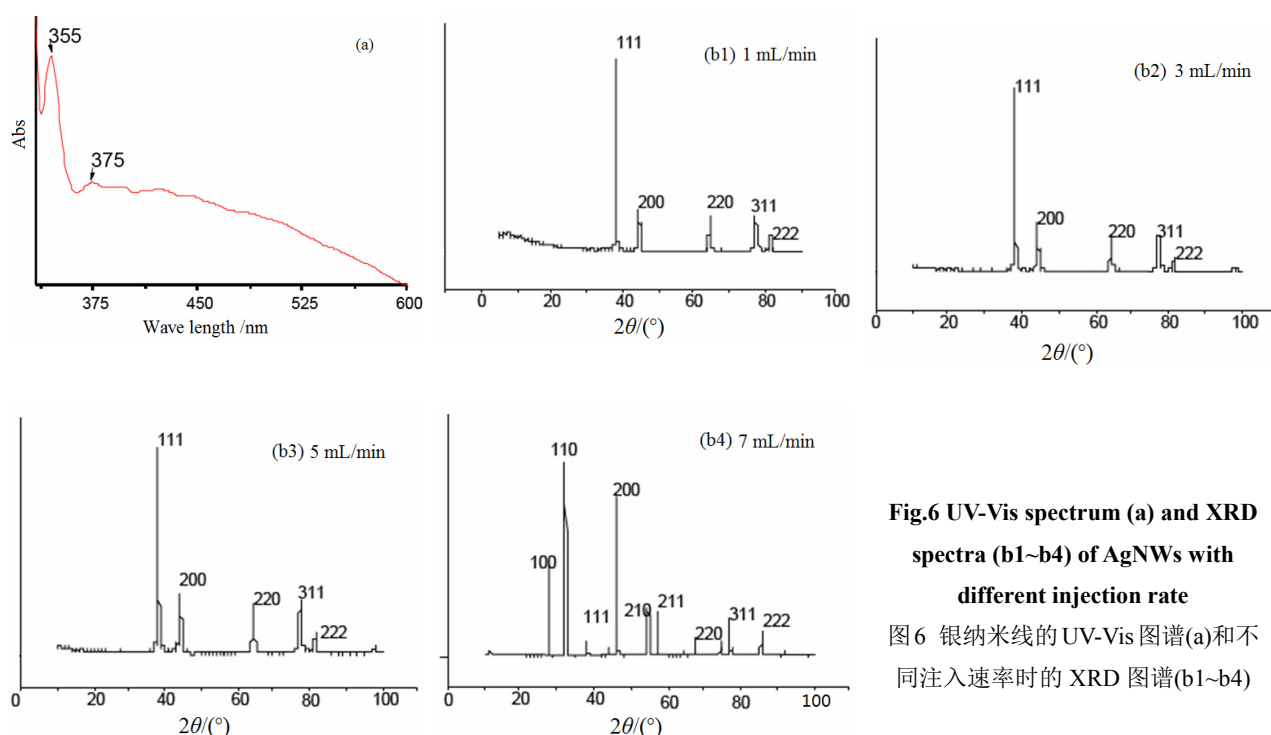


Fig.6 UV-Vis spectrum (a) and XRD spectra (b1~b4) of AgNWs with different injection rate

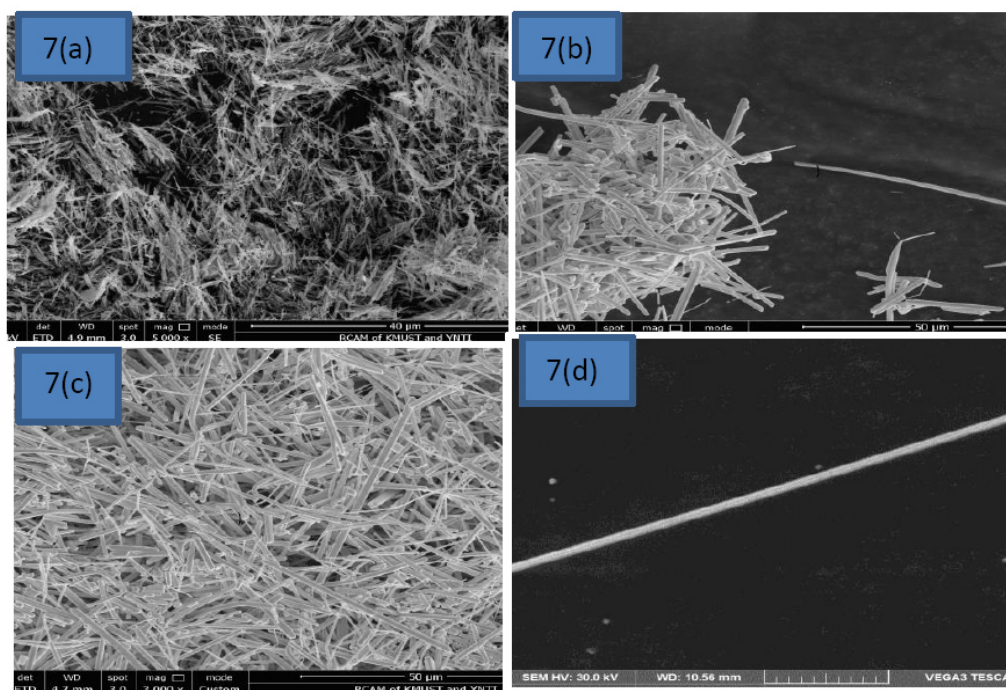
图6 银纳米线的UV-Vis图谱(a)和不同注入速率时的XRD图谱(b1~b4)

Fig.6(b) shows the XRD patterns of three different injection rates of AgNWs (1, 3 and 5 mL/min) indicated five diffraction peaks that were observed at $2\theta = 39.1^\circ$, 45.2° , 65.1° , 77.4° and 82° corresponding to the planes of the face-centered cubic (fcc) Ag nanostructures (111, 200, 220, 311 and 222), respectively; and injection rate (7 mL/min) shows more peaks than the other three (JCPDS File No. 04-0783). It has the lattice constant (a) 0.406634 nm and it is nearly closed to the literature value of lattice constant (0.4086 nm). Otherwise, XRD peaks of the AgNWs (3, 5, 7 mL/min) show weaker peak intensities than XRD peaks of AgNWs (1 mL/min) in which demonstrating that the synthesized AgNWs

have high crystallinity and also have the highest intensity of diffraction peak (111). Compared with the different injection rates (1, 3, 5 and 7 mL/min) of synthesized AgNWs pattern of fcc Ag, a high intensity of (111) reflection peak was shown in the X-ray diffraction pattern, and also pointing that the Ag particles have a specific (111) direction of the preferential lattice orientation. The intense peaks in X-ray diffraction pattern are also depended upon the orientation of crystal lattice side and crystal structure. It is illustrated that the peak intense at 39.1° which is corresponding to (111) plane that is higher intensity than the other peaks. It is indicating that AgNWs have apparent (111) orientation. (200) and (220) surfaces

have different electronic structures from (111) surface of fcc-structured metal nanoparticles. Therefore some crystallographic structure of silver metal chemically was interacted and changed to anisotropic shape with stabilize silver nanostructure^[15]. The highest intensity of (111) peak also illustrates along the (111) direction with apparent orientation growth of face centered cubic of AgNWs, it is also confirmed by the SEM/TEM. The X-ray diffraction pattern demonstrates that there is no orientation effects in the sample, while the highest intensity confirmed with those reported^[16-17]. AgNWs with without any further thermal treatment

which is in crystalline form were be indicated by using the observed reflection of the prepared AgNWs sample. To demonstrate the specific ways in which the structure of Ag NWs was indicated in the specific way which was also affected the scattering of light and absorption by employing UV-Vis spectroscopy and then electron microscopy were be used by analyzing the same materials. The shape and size of the AgNWs fundamentally were be determined by using the UV-Vis spectrum in which different frequencies that shows the corresponding different SPR bands^[18].



(a). 24000 g/mol; (b). 58000 g/mol; (c). 360000 g/mol; (d). 1300000 g/mol

Fig.7 SEM images of PVP molecular weight (M_w) effect on the diameter and length of AgNWs

图 7 PVP 平均分子量(M_w)对银纳米线直径和长度影响的 SEM 图像

2.2 Preparation of silver ink for transparent conductive film

The lamination coating process was used for the fabrication of AgNWs film on a PET substrate which was shown in Fig.8(b). Industrial laboratories used the lamination-assisted solution method that is coating technique for the controlled manner and making thin film. Lamination-assisted solution method in which the liquid was coated effectively and was adapted to higher throughput methods, more controllable method

like roll-to roll coating, slide coating and slot coating^[12]. And then spin-coating process that is the coating technique was used for the fabrication of silver nanowire film on a glass substrate as shown in Fig.8(c) and also used for making thin film. PET and glass substrate in which without coating material, or without any pre-treatment of hydrophobic or hydrophilic of PET and glass substrate were be used for the fabricated thin film like that the suspension of AgNWs coated onto them directly and uniformly and didn't

need to use any surfactants. In addition, the most important material of the uniformity of film is solvents. So AgNWs were dispersed in isopropanol, methanol, ethylene glycol, surfactants and distilled water. And then it placed in the ultrasonic bath in 60 min for sonication and put it in 5 h for completely agglomerated all of these suspensions. Otherwise, AgNWs have good dispersion ability and also have harder re-dispersion in aqueous solution than isopropyl alcohol or methanol. Therefore, isopropyl alcohol used for the more uniform coating on the films. In this work, we were used the article that came from the all of the suspensions of AgNWs in isopropyl alcohol. Laminating coating and spin coating processes used to get the uniformity of the films and then also determined by the film homogeneity in which interactions between substrates and nanomaterial, dispersion ability of suspension, concentration and solvents evaporation. In fact, we used isopropyl alcohol that has good evaporation, If the AgNWs/isopropyl alcohol concentration reached above 5 mg/mL, the film will show line recession.

AgNWs thin film surfaces are easily to oxidize that the fabricated original silver nanowire thin film with the film transmittance is less than 30% are non-conductive (R greater than 20 M Ω) because of its surface oxidation. Its surface oxidation took place due to the films drying process and coating process at room temperature. In this work, the oxidation on the silver nanowire surface was be dwindled by employing the concentrated hydrochloric acid. One of silver nanowire thin film on glass substrate and PET substrate put to incubate in the hydrochloric acid vapor (25 to 50°C) until 15 min and then the R value (resistance) gradually reduced to under 600 Ω .

The oxidation layers on the silver nanowire surface were be destroyed by employing the concentrated hydrochloric acid for improve the conductivity of silver nanowire film. During the weak adhesion to glass substrate and PET substrate, the prepared silver nanowire films were scratched and damaged easily. The sheet resistivity and transparency of the thin film are inversely proportional to the rise of the film thickness. The thin film electrodes have good

flexibility that can be bending nearly circle. Using the hydrochloric acid for increasing the contacts between nanowires and destroying the oxidized layer on the nanowire surfaces and then increased the film transparency and conductivity. In this process, hydrochloric acid reacted with the silver oxide to produce silver chloride which was slightly photo decomposed under the light illumination. In this experiment, the main impurities species is chloride. Therefore small amount of chlorine presented in all sample. (Cl-K at nearly 2.65 keV)^[20]. So junction resistance between the AgNWs is greater than 1 G Ω . The nanowire-nanowire junction resistance and the single nanowire junction resistance are increased while the silver nanowire surface is oxidized due to the silver oxide's semiconducting property. Fig.8(a) shows the transmittance percent of silver nanowire thin film on glass substrate (80%) and PET substrate (88%) and also shows surface resistivity of AgNWs thin film on glass substrate (26 Ω /sq) and PET substrate (17 Ω /sq) respectively. According to the transmission spectra, there are no any distinct absorption peaks of silver nanowire exception that periodic intensity oscillation that is a sign for the reflection light of uniform thin film. The scattering and reflection of light on silver nanowire film is increased and the film transmittance is decreased because of the diameter of AgNWs are larger than 100 nm. AgNWs with a wide range of aspect ratios from 350 to greater than 3000 were prepared by controlling various reaction parameters. Three types of AgNWs with a large difference in aspect ratio were used either separately or together to investigate the performance characteristics of conductive ink. First, the film coated with AgNWs having small diameter and length I (30 nm, 30 μ m) and II (60 nm, 50 μ m) of PET and glass substrate showed a transmittance of 88%, 83%, 82%, 76% and resistivity of 1m Ω /sq, 0.03, 8 and 3 m Ω /sq respectively. The surface resistivity of this film was too high to be used for conductive devices even though the transmittance was suitable for the transparent electrodes in a display panel. Scattering and reflection apply the application of silver nanowire thin films in photovoltaic devices, in which the silver

nanowire electrode consisted of solar cells display greater photocurrent than the application of ITO film electrode. In some electronic devices, thin film which made from small diameter of AgNWs display more transparency at same conductivity than other^[21]. On the other hand, thin film coated of PET and glass substrate with AgNWs having long length 3# (100 nm, 70 μm) exhibited a transmittance of 77%, 70% and resistivity of 56, 27 $\text{m}\Omega/\text{sq}$ respectively. In this case, long length of AgNWs exhibited that decreased the

transmittance and the resistivity. And then thin film of PET have more transmittance/% than glass substrate and thin film of glass substrate also have high resistivity than it. Then mixture of AgNWs with high and low aspect ratio were used and the thin film of PET has good flexibility and can also be bending like circle and thin film of glass substrate have less flexibility. Therefore, thin film of PET is more suitable used for flexible transparent electrode than the thin film of glass substrate.

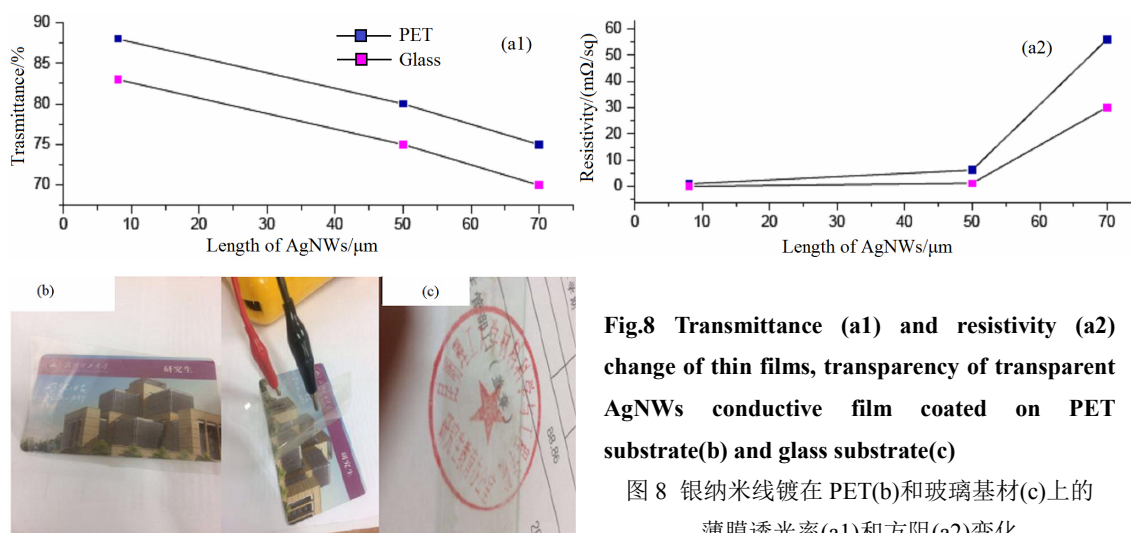


Fig.8 Transmittance (a1) and resistivity (a2) change of thin films, transparency of transparent AgNWs conductive film coated on PET substrate(b) and glass substrate(c)

图 8 银纳米线镀在 PET(b)和玻璃基材(c)上的薄膜透光率(a1)和方阻(a2)变化

Fig.8 shows that the symbols of Kunming University of Science and Technology was clearly seen through transparent conductive film coated with 99% AgNWs with small aspect ratio I (30 nm, 30 μm) and the surface resistivity of thin-film PET and glass substrate are 17 and 26 Ω/sq and transmittable percent are 88% and 80%. These results indicate that AgNWs prepared in this study can be used as an alternative ITO.

3 Conclusion

The present work shows that AgNWs could be synthesized using the polyol process. The average diameters (17~100 nm) of AgNWs were synthesized by using polyol method. This research demonstrated characterization of AgNWs and confirmed that the sample D of AgNWs has more uniform diameter and less impurity than others and then continuously

studied it with different injection rates in which sample D with injection rate (1 mL/min) is more longer length than the other injection rates. Then larger molecular weight (M_w) of PVP is produced m longer length of AgNWs (above 100 nm). SEM images, TEM images and XRD spectra confirmed that more uniform silver nanowire. Fig.6(a) shows the absorption spectra of AgNWs synthesized with different molar of AgNO_3 . The appearance of a story peak appeared at 355 nm that can be determined as the transverse mode of relatively long AgNWs. Four point probe method used for detection of resistivity and resistance of thin films. In this report, the diameter and length of AgNWs were controlled by changing the AgNO_3/PVP molar ratio, injection rate and M_w of PVP. We demonstrated that the injection rate and larger M_w of PVP are the most important to control the diameter and length of silver nanowire by using the polyol method and it also used for the prepared AgNWs with

a long length (30 nm to above 100 μm) and thin diameter (17~100 nm). The fabrication of transparent conductive films of PET and glass substrate using a scalable lamination-assisted solution method and spin-coating process is described in this study. For transparent conductive film of PET and glass substrate, silver nanowire inks with a resistance of 17, 26 Ω/sq and transmittance of 88%, 80% respectively were produced using a mixture of AgNWs with high and low aspect ratio. Thin film of PET is more suitable used for flexible transparent electrode than thin film of glass substrate. The use of AgNWs thin film could add extra functionality and future work will involve testing them for applications.

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