



# Influence of Ag Nanowires with Different Morphologies on Light Trapping Abilities and Optoelectronic Properties of Ag Nanowires/ZnO:Al Nanorods Composite Films

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Abstract: The Ag nanowires/ZnO:Al nanorods (Ag NWs/AZO NRs) composite films were prepared by the simple hydrothermal growth of AZO NRs on the AZO seed layer (SL) at the void regions among the Ag NWs. Distinctive morphology evolution of the Ag NWs depending on the mass of FeCl<sub>3</sub>·6H<sub>2</sub>O solution was observed on the AZO SL. The effect of Ag NWs with different morphologies on the structure, morphology, optoelectronic properties and light trapping abilities of Ag NWs/AZO NRs composite films was investigated systematically. In particular, the relationship between the morphology, light trapping and electrical properties of the composite films was analyzed in detail. When 7 g of FeCl<sub>3</sub>·6H<sub>2</sub>O solution was added, Ag NWs with a length of about 50  $\mu$ M were generated, and the Ag NWs overlapped adequately with each other to form a network structure beneficial to conductivity. Meanwhile, the Ag NWs/AZO NRs composite films containing Ag NWs prepared with 7 g FeCl<sub>3</sub>·6H<sub>2</sub>O solution exhibited high TT (above 80%), high haze value (0.29) at 550 nm and low sheet resistance (5.9  $\Omega$ /sq), which can be employed as transparent electrodes for improving electrical and light trapping properties in solar cells.

Keywords: ZnO:Al; Ag nanowires; morphology; light trapping abilities; optoelectronic properties; haze

## 1. Introduction

Transparent conducting oxide (TCO) films such as SnO<sub>2</sub>:F (FTO), In<sub>2</sub>O<sub>3</sub>:Sn (ITO) and ZnO:Al (AZO) films have been widely applied in various optoelectronic devices due to their excellent optoelectronic properties [1–3]. Among these TCO films, AZO films, as front electrodes in solar cells, have been gaining attention recently because of their good optoelectronic properties, low cost, non-toxicity, and stability under a hydrogen plasma environment [4–6].

As front electrodes, AZO films with excellent light trapping capabilities and optoelectronic properties can effectively improve the conversion efficiency of thin film solar cells. In recent years, in order to obtain excellent light trapping capabilities, AZO films with textured structures have been researched [7,8]. Single-textured films have been prepared by plasma etching after sputtering deposition [9], and crater-crater-like double-textured films have been prepared by multiple sputtering and wet chemical etching processes [10,11]. However, these methods have disadvantages such as high requirements for experimental equipment and complex process of preparation. Single-textured AZO films with nanorods (NRs) structure have also been prepared by electrochemical deposition and hydrothermal methods, etc. [12,13]. The hydrothermal method has the advantages of being simple and having effective control of crystal growth. Recently, some studies have shown that Ag nanowires (NWs) can provide proper light trapping and electrical capabilities [14-16]. Therefore, we expect that by introducing Ag NWs into AZO NRs to form Ag nanowires/AZO nanorods (Ag NWs/AZO NRs) composite films, the light trapping and electrical properties can be further enhanced simultaneously. It is well known that the morphology changes of Ag NWs prepared by polyol methods are closely related to the growth conditions, such as



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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). the mass of the added shape-controlling agent, the temperature and time of the reaction, the molar ratio of PVP to AgNO<sub>3</sub>, and the concentration of AgNO<sub>3</sub> [17–20]. Different morphologies of Ag NWs will affect the light trapping abilities and electrical properties of Ag NWs/AZO NRs composite films. However, to the best of our knowledge, the effect of Ag NWs with different morphologies on the light trapping abilities and electrical properties of Ag NWs /AZO NRs composite films has not been investigated so far.

In this paper, we prepared Ag NWs/AZO NRs composite films, which exhibited excellent electrical and light trapping properties, using the simple hydrothermal growth of AZO NRs on the AZO seed layer (SL) at the void regions among the Ag NWs. Ag NWs with different morphologies prepared by adding FeCl<sub>3</sub>·6H<sub>2</sub>O solution with different masses were obtained. Additionally, the influence of Ag NWs with different morphologies on the structure, morphology, optoelectronic properties and light trapping capabilities of Ag NWs/AZO NRs composite films was demonstrated systematically.

#### 2. Experimental Method

Three-hundred nanometer-thick AZO SL was fabricated on a glass substrate by direct current pulsed magnetron sputtering. During the sputtering deposition process, ZnO:Al<sub>2</sub>O<sub>3</sub> (98 wt%:2 wt%) was utilized as the target material and Ar was employed as the sputtering gas, and the sputtering power and working pressure were set at 0.8 Pa and 350 W, respectively.

Ag NWs were prepared by the polyol method. 0.53 g of PVP (Aladdin, Shanghai, China, K90) with 0.41 g of AgNO<sub>3</sub> (Sinopharm, Shanghai, China, AR) was added to 40 mL of ethylene glycol (EG, Sinopharm, Shanghai, China, AR) and stirred magnetically at 60 °C for 1 h until complete dissolution. Then, 1 g, 3 g, 7 g, and 9 g of FeCl<sub>3</sub>·6H<sub>2</sub>O (Sinopharm, Shanghai, China, AR) solutions (600  $\mu$ M in EG) were added. Four mixed solutions containing different masses of FeCl<sub>3</sub>·6H<sub>2</sub>O solutions were quickly injected into Teflon-lined autoclaves that were then sealed and placed in an oven for heating at 130 °C for 5 h. After the reaction was completed, the Ag NWs precipitate was obtained by centrifugation and washed with acetone (Xilong Scientific, Shantou, China, AR) and anhydrous ethanol (Sinopharm, Shanghai, China, AR), respectively. Finally, the obtained Ag NWs were dispersed in 50 mL of anhydrous ethanol for the subsequent process.

Ag NWs films were fabricated by the spin-coating technique. The well-dispersed Ag NWs solution was spin-coated 4 times on the AZO SL at 3000 rpm for 20 s. Then, the spin-coated films were annealed for 1 h in a tube furnace under vacuum at 250 °C. Ag NWs films prepared on the AZO SL with 1 g, 3 g, 7 g, and 9 g of FeCl<sub>3</sub>.6H<sub>2</sub>O solutions were obtained and denoted as samples Ag1, Ag2, Ag3, and Ag4, respectively.

Last, Ag NWs/AZO NRs composite films were prepared by the hydrothermal method based on the exposed AZO SL under the Ag NWs.  $Zn(CH_3COO)_2H_2O$  (Sinopharm, Shanghai, China, AR) with  $C_6H_{12}N_4$  (Sinopharm, Shanghai, China, AR) aqueous solution at equal concentrations of 0.08 M was used as precursor solution at room temperature. Then,  $Al(NO_3)_3.9H_2O$  (Sinopharm, Shanghai, China, AR) with a [Al]/[Al + Zn] ratio of 3% was employed as the doping source added to the aqueous solution. The mixture solutions were poured into Teflon-lined autoclave apparatus containing samples Ag1, Ag2, Ag3, and Ag4 for hydrothermal growth in an oven at 90 °C for 3.5 h to form Ag NWs/AZO NRs composite films (Ag1-AZO, Ag2-AZO, Ag3-AZO and Ag4-AZO).

The surface morphologies of the films were investigated by field-emission scanning electron microscopy (FESEM, SIGMA HD, Zeiss, Germany). The X-ray diffraction (XRD, X'Pert, PANalytical, The Netherlands) was used to analyze the crystal structures of the films. The sheet resistance of the films was measured using an RTS-8 four-point probe instrument. Total transmittance (TT) and specular transmittance (ST) spectral measurements of the films were performed by a U-3310 ultraviolet-visible spectrophotometer with an integrating sphere. Diffuse transmittance (DT) was calculated as DT = TT - ST. Additionally, the haze spectra were determined by TT and DT spectra (Haze = DT/TT).

## 3. Results and Discussion

## 3.1. Structural Properties

A comparison of XRD patterns of the films before and after the growth of AZO NRs is shown in Figure 1a. It can be seen from Figure 1a that all the films show a strong and sharp diffraction peak at 20 of about 34.4°, corresponding to the (002) plane, and an insignificant peak near 72.5°, corresponding to the (004) plane (JCPDS No. 36-1451). Additionally, the intensity of the (002) peak is increased after the growth of AZO NRs. The results indicate that all AZO NRs in the composite films have a hexagon wurtzite structure and c-axis orientation [21]. Meanwhile, there are no characteristic peaks associated with  $Al_2O_3$  in the XRD pattern. XRD patterns of all the Ag NWs/AZO NRs composite films and the corresponding partially enlarged patterns in the same  $2\theta$  degree range are shown in Figure 1b. As shown by the corresponding partially enlarged patterns, two diffraction peaks appear gradually at about 38.1° and 44.6°, corresponding to the (111) and (200) crystal plane of Ag (JCPDS No. 04-0783), respectively. In addition, from sample Ag1-AZO to Ag4-AZO, two peaks gradually increase in intensity, which indicates the formation and increase in length of Ag NWs. Furthermore, it can be seen from Figure 1b that the intensity of the (002) peak is gradually decreased from sample Ag1-AZO toAg4-AZO. This may be caused by the variation in the length of Ag NWs. As the mass of the added FeCl<sub>3</sub>·6H<sub>2</sub>O solution increases, Ag NWs begin to be generated and gradually increase in length, resulting in the increased coverage of Ag NWs on AZO SL and the reduced area of exposed AZO SL. Thus, the number of AZO NRs grown based on AZO SL during the hydrothermal process decreases accordingly, leading to the decrease in the intensity of the (002) peak. The above results can also be confirmed by the following SEM images.



**Figure 1.** (a) Comparison of XRD patterns of the films before and after the growth of AZO NRs; (b) XRD patterns of all the Ag NWs/AZO NRs composite films.

#### 3.2. Surface Morphology

Figure 2 shows the surface SEM images of Ag NWs films on the AZO SL (Ag1, Ag2, Ag3, and Ag4) and Ag NWs/AZO NRs composite films (Ag1-AZO, Ag2-AZO, Ag3-AZO, and Ag4-AZO). It can be clearly seen that adding different masses of the FeCl<sub>3</sub>·6H<sub>2</sub>O solution has a significant effect on the morphology changes in the Ag NWs films on the AZO SL, as depicted in Figure 2a,c,e,g. For sample Ag1, when 1 g of the FeCl<sub>3</sub>·6H<sub>2</sub>O solution is added, some randomly arranged Ag nanoparticles appear on the AZO SL, and more AZO SL area is exposed. FeCl<sub>3</sub> affects the yield of Ag and the formation of Ag NWs [17]. Shorter Ag NWs with an average length of 5  $\mu$ M are generated, and only a small number of Ag NWs overlap each other, while the mass of the FeCl<sub>3</sub>·6H<sub>2</sub>O solution raises to 7 g (sample Ag3), the length of Ag NWs increases to about 50  $\mu$ M and more Ag NWs

can be connected, which results in a reduction in the exposed AZO SL area. When the mass of the FeCl<sub>3</sub>·6H<sub>2</sub>O solution increases to 9 g (sample Ag4), the length of Ag NWs continues to increase, and a large quantity of Ag NWs densely overlap, making most of the AZO SL covered. From the above results, it is obvious that with the increase in the added FeCl<sub>3</sub>·6H<sub>2</sub>O solution from 1 to 9 g, the shape of the generated Ag transforms from nanoparticles to NWs, and the length of Ag NWs gradually increases, causing them to more adequately overlap with each other. This may be due to the fact that Fe<sup>3+</sup> limits the number of nuclei of multiply twinned particles (MTPs). During the polyol process, as the mass of the FeCl<sub>3</sub>·6H<sub>2</sub>O solution increases in a certain range, the amount of free Fe<sup>3+</sup> grows in the reaction solution, which reduces the number of nuclei of the generated MTPs, and fewer seeds allow more adequate growth of Ag NWs [17]. This also corresponds to the increase in the (111) and (200) peak intensities shown in the XRD results.



**Figure 2.** Top-view SEM images of the Ag NWs films on the AZO SL: (**a**) Ag1; (**c**) Ag2; (**e**) Ag3; (**g**) Ag4. The Ag NWs/AZO NRs composite films: (**b**) Ag1-AZO; (**d**) Ag2-AZO; (**f**) Ag3-AZO; (**h**) Ag4-AZO.

It can also be seen clearly that Ag NWs with different morphologies have a significant effect on the morphology changes in the Ag NWs/AZO NRs composite films, as depicted in Figure 2b,d,f,h. For sample Ag1-AZO, it can be seen that the AZO NRs grown using the hydrothermal method are closely aligned with each other, and the surface of the composite films is relatively dense. Meanwhile, Ag NWs are not observed on the surface of the composite films. Subsequently, for sample Ag2-AZO, the AZO NRs are fully grown in the void region among the Ag NWs on the AZO SL, and the composite structure with intertwined Ag NWs and AZO NRs starts to appear on the surface of the composite films. The number of AZO NRs begins to decrease because of the existence of Ag NWs among the AZO NRs, which corresponds to the decrease in the (002) peak's intensity in the XRD results. Then, for samples Ag3-AZO and Ag4-AZO, the composite structure with intertwined Ag NWs and AZO NRs could still be observed. In addition, it can also be observed that some Ag NWs are not completely wrapped by AZO NRs, which is due to the increase in the overlap density of Ag NWs and the limited growth height of AZO NRs, so more Ag NWs with higher overlap heights are not wrapped by AZO NRs. The changes in the morphology resulting from the length of Ag NWs could affect the light trapping capabilities of the films, which will be confirmed by the following studies of haze.

#### 3.3. Optical Properties

The optical properties of all the films are investigated. The TT and DT spectra of Ag NWs films on the AZO SL and Ag NWs/AZO NRs composite films are shown in Figure 3a,b, respectively. It can be observed that for samples Ag1, Ag2, Ag3, and Ag4, in the visible wavelength range (400–800 nm), the average TT of Ag NWs films on the AZO SL is 81.8%, 80.5%, 74.7%, and 69.8%, and for samples Ag1-AZO, Ag2-AZO, Ag3-AZO, and Ag4-AZO, the average TT of Ag NWs/AZO NRs composite films is 81.5%, 81.2%, 75.2%, and 65.8%, respectively. The average TT of the Ag NWs and Ag NWs/AZO NRs films gradually decreases as the mass of the  $FeCl_3 \cdot 6H_2O$  solution used during the preparation of Ag NWs increases, which may be caused by the increase in the length and lap density of Ag NWs. Except for the sample Ag4-AZO, the average TT of all other composite films is higher than 80% (excluding the effect of the glass substrate with a transmittance of about 90%), which is appropriate for application in the front electrodes of solar cells. The average TT of Ag NWs/AZO NRs and Ag NWs films is not significantly different under the same preparation conditions of Ag NWs, indicating that the AZO NRs grown on the AZO SL at the void regions among the Ag NWs have no significant impact on the TT of the films. In addition, it can also be seen that the DT of the films gradually increases from Ag1 to Ag4 and Ag1-AZO to Ag4-AZO, which will determine the light trapping capability of the films.



**Figure 3.** TT and DT spectra of (**a**) Ag NWs films on the AZO SL and (**b**) Ag NWs/AZO NRs composite films.

#### 3.4. Light Trapping Ability

In order to investigate the relationship between the light trapping capability and surface morphology of all the films, the haze spectra of the films are measured (Haze = DT/TT). From Figure 4a,b, it can be seen that the haze values of both Ag NWs films on the AZO SL and Ag NWs/AZO NRs composite films gradually increase with the increase in the amount of FeCl<sub>3</sub>·6H<sub>2</sub>O solution added during the preparation of Ag NWs, which means that the light trapping capability of the films is closely related to the morphological changes in Ag NWs and the composite films. From the above SEM results, as shown in Figure 2a,c,e,g, as the length of Ag NWs gradually increases, the lap density between Ag NWs also becomes larger, which causes a decrease in the size of the mesh among the Ag NWs, leading to an increase in light scattering and a corresponding gradual increase in the haze of the films [22]. According to the SEM results previously mentioned in Figure 2b, for sample Ag1-AZO, Ag NWs are not generated, and the AZO NRs are closely aligned with each other, which results in a lower haze value (0.17) of the films at 550 nm [23]. Additionally, for samples Ag2-AZO-Ag4-AZO, with the increase in the length of Ag NWs, the Ag NWs and AZO NRs on the surface of the films intertwine with each other, and gaps start to appear among the AZO NRs. The appearance of the composite structure increases the surface roughness of the films, which causes more scattering of the incident light and improves the light trapping ability of the films [24]. The haze value of the sample Ag3-AZO reaches 0.29 at 550 nm, which indicates that the films have excellent light trapping capabilities. Although the sample Ag4-AZO has a higher haze value, it is not suitable for the solar cell front electrode application due to its low TT.



Figure 4. Haze spectra of (a) Ag NWs films on the AZO SL and (b) Ag NWs/AZO NRs composite films.

#### 3.5. Electrical Properties

The electrical properties of all the films are analyzed by measuring the sheet resistance, as shown in Figure 5. The sheet resistance of AZO SL film is about 33.9  $\Omega$ /sq. From Figure 5, we can see that the sheet resistance of samples Ag1 and Ag1-AZO is about 33.6  $\Omega$ /sq and 95.6  $\Omega$ /sq, respectively. Additionally, as the mass of the added FeCl<sub>3</sub>·6H<sub>2</sub>O solution increases to 3 g, the sheet resistance of samples Ag2 and Ag2-AZO has not changed significantly. It can be found that the electrical properties of the Ag NWs films on the AZO SL are not enhanced compared to the AZO SL films, which is because the Ag nanoparticles and short Ag NWs on the AZO SL are not connected to form effective conductive networks. While the sheet resistance is decreased evidently for the films prepared with the mass of FeCl<sub>3</sub>·6H<sub>2</sub>O solution from 7 to 9 g, which is accounted for by the increase in the length and lap density of Ag NWs, resulting in the formation of more conductive networks among Ag NWs. The sheet resistance of the sample Ag3-AZO is 5.9  $\Omega$ /sq, indicating that the composite films have excellent electrical conductivity. The Ag NWs/AZO NRs composite films of sample Ag3-AZO have high transmittance, and both the light trapping and electrical properties are improved significantly.



Figure 5. Sheet resistances of Ag NWs films on the AZO SL and Ag NWs/AZO NRs composite films.

#### 4. Conclusions

In general, the Ag NWs/AZO NRs composite films were prepared by the simple hydrothermal growth of AZO NRs on the AZO SL at the void regions among the Ag NWs. XRD patterns confirmed the formation of Ag NWs and AZO NRs in the composite films, and AZO NRs had a hexagon wurtzite structure and c-axis orientation. As the mass of the added  $FeCl_3 \cdot 6H_2O$  solution increased from 1 to 9 g, the shape of the generated Ag transformed from nanoparticles to NWs, and the length of Ag NWs gradually increased. The composite structure of interlaced AZO NRs and Ag NWs gradually formed on the surface of the AZO SL films. The average TT in the visible wavelength range of all other composite films was higher than 80%, except for the sample Ag4-AZO. The light trapping abilities and electrical properties of the composite films were gradually enhanced with the increase in the length of Ag NWs. It was worth noting that by introducing Ag NWs into AZO NRs, the light trapping abilities and electrical properties of the Ag NWs/AZO NRs composite films can be improved simultaneously. In addition, the Ag NWs/AZO NRs composite films of sample Ag3-AZO exhibited high TT (above 80%), low sheet resistance (5.9  $\Omega$ /sq) and high haze value (0.29) at 550 nm. The results show that the Ag NWs/AZO NRs composite films have satisfactory optoelectronic properties and light trapping abilities, which are beneficial to their application in front electrodes of solar cells.

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