Jurnal Penelitian Fisika dan Aplikasinya (JPFA)
p-ISSN: 2087-9946
e-ISSN: 2477-1775

Volume 09, Issue 01, June 2019
DOI: 10.26740/jpfa.v9n1.p1-9
http://journal.unesa.ac.id/index.php/jpfa

Research Article

Silver Nanorods Layer Based on Polyvinyl Alcohol on Glass Substrates by Dip-Coating Method

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Abstract

This research reports the investigation of the performance of a thin layer based on silver nanorods using the dip-coating method. The synthesis was conducted by polyol method at an oil bath temperature of 140 °C. In the synthesis of silver nanorods, materials used were silver nitrate ($AgNO_3$) as the main raw material, ethylene glycol (EG) as the solvent, and a small amount of sodium chloride (NaCl) as a mediated-agent (precursor). Polyvinyl alcohol (PVA) used as a capping agent and stabilizer in this process. Diameter and length of silver nanorods were 800 nm and 15 µm, respectively. Furthermore, the silver nanorods suspension was deposition onto a glass substrate with a variety of dipping cycles. The result showed the thickness of the thin layer is linear with a number of dipping cycles. Electrical and optical properties of thin layer show that sheet resistance about of 30 Ω sq^{-1} by transmittance above of 80%. The silver nanorods thin film can be used as a conductive and transparent electrode for various optoelectronic applications.

Keywords: *silver nanorods, transparent electrode, optical properties, sheet resistance, dip-coating*

Lapisan Tipis *Silver Nanorods* Berbasis Polivinil Alkohol pada Subtrat Kaca dengan Metode *Dip Coating*

Abstrak

Pada penelitian ini, kami telah menyelidiki kinerja dari lapisan tipis berbasis silver nanorods menggunakan metode dip-coating. Sintesis dilakukan dengan metode poliol dalam media oil bath pada suhu 140 °C. Dalam sintesis silver nanorods, bahan yang digunakan adalah perak nitrat ($AgNO_3$) sebagai bahan baku utama, etilen glikol (EG) sebagai pelarut, dan sedikit natrium klorida (NaCl) sebagai agen mediasi (precursor). Polivinil alkohol (PVA) digunakan sebagai capping agent dan penstabil dalam proses ini. Diameter dan panjang silver nanorods diperoleh masing-masing 800 nm dan 15 µm. Selanjutnya, suspensi silver nanorods dilapiskan di atas substrat kaca dengan variasi jumlah celupan. Hasil penelitian menunjukkan bahwa ketebalan lapisan tipis adalah linier dengan jumlah pencelupan.

Sifat listrik dan optik dari lapisan tipis menunjukkan bahwa hambatan dari lembaran diperoleh sekitar 30 Ω sq^{-1} dengan transmitansi di atas 80%. Film tipis silver nanorods dapat digunakan sebagai elektroda konduktif dan transparan untuk berbagai aplikasi piranti optoelektronika.
Kata Kunci: *silver nanorods, elektroda transparan, sifat optik, resistansi lembar, dip-coating*

PACS: 78.67.Uh, 78.67.Qa, 81.07.Gf, 81.15.Rs, 07.05.Dz

Article History: Received: September 17, 2018 Decided to resubmit (Round 1): January 3, 2019
Revised (Round 1): February 4, 2019 Approved with minor revision: May 13, 2019
Accepted: June 27, 2019 Published: June 30, 2019

How to Cite: Junaidi, Riyanto A, Triyana K, and Khairurrijal. Silver Nanorods Layer Based on Polyvinyl Alcohol on Glass Substrates by Dip-Coating Method. *Jurnal Penelitian Fisika dan Aplikasinya (JPFA)*. 2019; **9**(1): 1-9. DOI: https://doi.org/10.26740/jpfa.v9n1.p1-9.

I. INTRODUCTION

The number of electronic devices for a display and touch screens has increased significantly in the last decade [1]. The transparent electrode can be used for various electronic devices, such as organic solar cells, displays, touch screens, and transparent heaters. Inorganic solar cells, the transparent electrode is used for anode or cathode to produce a charge carrier. It is believed that the transparent electrodes have several advantages, i.e. flexible substrate, easy to fabrication, low cost, and lightweight. The widest materials used for the transparent electrodes are metal oxides of indium tin oxide (ITO). ITO is a highly transparent electrode with the transmittance (T = 80-95 %) and low sheet resistance (Rs = 10-1000 Ω sq^{-1}) [2-3].

ITO, however, cannot cover all qualifications thanthe expected as a transparent electrode. ITO has some weaknessessuch as (a) ITO by nature has the ceramics properties (not flexible), easy to crack because of the low strain and have a rapid decline in the conductivity, (b) indium is a rare material, so it is very expensive, (c) the process to make ITO is very expensive as it requires high temperature and vacuum to control the thickness, (d) ITO should kept in a special place in order to avoid any damages of the base layer organic devices, and (e) ITO has high refractive index which is not suitable for display applications because can be to reflect light and reduce the brightness of the screen [4-5].

Several researches have been conducted to solve problems associated with the transparent electrode of ITO. For example, ITO made a flexible electrode by drop-coated onto flexible substrates such as polyethylene terephthalate (PET). However, it does not only increase the production costs but also reduces the optical and electrical performance of an electrode. Therefore, many researchers have tried to find the alternative materials to replace ITO in the last decade. The materials used to substitute ITO include carbon nanotubes (CNTs), graphene, and metal nanorods or nanowires. The alternative materials have also been generated that it can apply as a substitute for ITO transparent electrode [5-8].

One of the main focuses of the research is the manufacture of a transparent electrode by using silver nanorods (AgNRs) or silver nanowires (AgNWs) [9]. Silver by nature is high electrical and thermal conductivity, transparency and stability [10-13]. The manufacture of a transparent electrode based on AgNWs and AgNRs was performed using Meyer-rod coating, spin coating, spray coating, dip-coating, brush-painting, electrospinning, and roll-to-roll process. The deposition process was carried out onto a transparent glass substrate, quartz glass, PET, and ITO. In the manufacture of a transparent electrode, the main attention focused on some important factors, such as conductivity (σ), resistance (R_s), the transmittance (T), and haze. Transparent electrode developed using silver nanorods or nanowires have sheet resistance of ~50 Ω sq^{-1}, transmittance above of 75 %, and the haze below of 5 % [14-21].

This study develops a transparent electrode based on silver nanorods. The synthesis of silver nanorods used polyvinyl alcohol (PVA) as a capping agent and stabilizer. PVA is a non-toxicity polymer, have good mechanical strength, electrochemical stability, low membrane permeability, and high crystallinity. Deposition process was carried out by assessing the effect of variations of dipping cycles to the optical and electrical conductivity of AgNRs thin layer. Furthermore, AgNRs thin layer characterized using a current-voltage (I-V meter), a voltage-resistance meter data acquisition system (DAS MA-16), a digital multimeter, a UV-vis spectrometer, and scanning electron microscope (SEM).

II. METHOD

Materials and preparation: Silver nitrate ($AgNO_3$, Merck), polyvinyl alcohol (PVA Mw. 31000-50000 g/mol, Sigma-Aldrich), sodium chloride (NaCl, 98 %, Merck), ethylene glycol (EG, 99 %, Merck), ethanol (EtOH, 98 %, Merck), deionized water, and glass substrate. Silver nanorods with an average diameter of approximately 800 nm and length of 10 to 20 μm were synthesized by the reduction of silver nitrate in the present of polyvinyl alcohol in ethylene glycol by polyol method.

The amount of ethylene glycol was heated at 140 °C and 700 rpm for 10 min. Furthermore, solution of $AgNO_3$/EG, NaCl/EG, and PVA/EG (0.5 M, 10 mM, 2 M) was injected for 0.5 cc/min. After the mixture for 2 hours, the resulting AgNRs suspension was separated and washed with ethanol by centrifugation at the rate of 6000 rpm for three times to remove supernatant.

Thin layer was fabricated using AgNRs suspensions by dip-coating method onto the glass substrate 10 x 25 mm^2. Glass substrates were cleaned with deionized water and ethanol for 30 min in an ultrasonic bath. Finally, the thin layer was dried in an oven at 200 °C for 10 min to remove the solvent.

Characterization of transparent electrodes: The electrical and optical properties of the transparent electrode were measured using a current-voltage (I-V meter, ELKAHFI-100), a voltage-resistance meter data acquisition system (V-R meter, DAQ MA-16), a digital multimeter (DT-850L), and a UV-vis spectrometer (Shimadzu, UV-1700). The hybrid electrodes on the glass substrate were observed using a field-emission scanning electron microscope (FE-SEM) (JEOL, JSM-6510LA) by accelerating the voltage of 10 kV.

III. RESULTS AND DISCUSSION

A transparent conductive electrode was produced using a simple dip-coating method. The silver nanorods deposition process was carried out onto a glass substrate 10 x 25 mm^2. The optical absorbance of AgNRs suspension was characterized using a UV-vis spectrophotometer in the wavelength range of 300 to 700 nm as shown in Figure 1.

The optical absorbance of AgNRs suspension obtained two absorbance peaks, namely at 380 nm and 390 nm. All peaks indicate that the optimum AgNRs was formed from the synthesis process. According to some references, the peak of the absorption spectrum of AgNRs is ranged from 350 to 390 nm [24, 25].

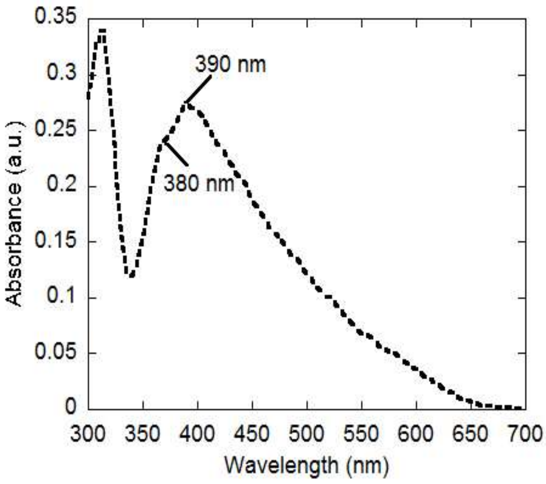

Figure 1. UV-vis Spectra of AgNRs Suspension

The dip-coating method is a simple method for deposition materials by dipping a substrate into AgNRs solution. The coating process of the substrate was repeated with variations in the number of dipping cycles. When the substrate was dipped and withdrawn from the AgNRs solution, then the number of AgNRs would be attached to the substrate with a solvent. To evaporate and eliminate the solvent, then the glass substrate was heated at a temperature of 200 °C for 10 minutes for each cycle. The thickness of the thin layer was highly dependent on the number of dipping cycles, the speed of cycles dipping, solvent vapor pressure, and the concentration of the AgNRs solution [19]. Diagram process for making the AgNRs layer by the dip-coating method can be illustrated in Figure 2(a-c).

Figure 2(d) shows the number of dipping cycles of AgNRs suspension deposition which was linear with the sheet resistance and inverse with the value of optical transmittance. When the coating process was carried out, the number of dipping cycles would continue to expand the number of AgNRs attached to the substrate. This results in the value of the optical transparency of the substrate would decrease according to the thickness of the AgNRs layer. The number of dipping cycles can increase the value of the sheet resistance of the AgNRs layer. The absence of built-up effect due to repeated dyeing process shows the AgNRs was coated strongly on the substrate [26].

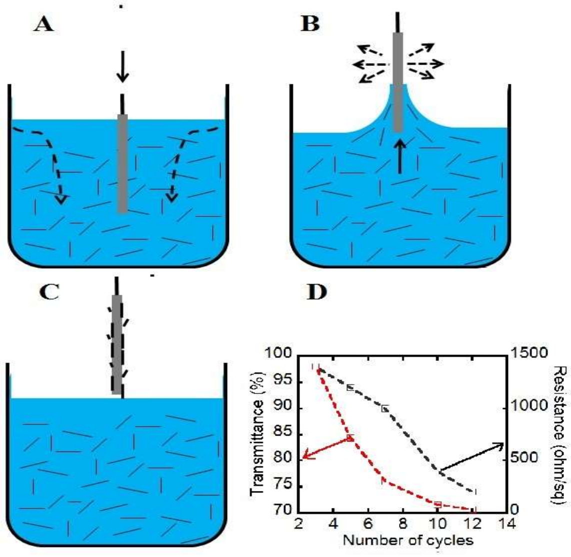

Figure 2(a-c). Illustration of the Workflow of Dip-Coating and (d) the Resulting Plot of the Transmittance and Resistance by the Number of Cycles

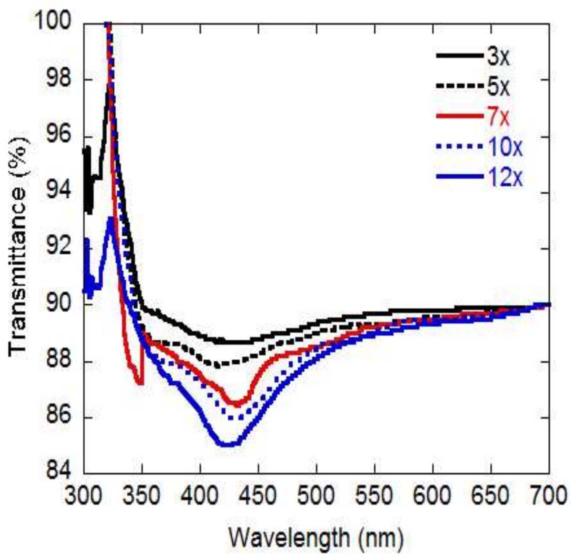

Figure 3. UV-vis Spectrometer of AgNRs Layer

Optical transmittance of the AgNRs layer is shown in Figure 3 and Figure 4(a) the optical transmittance was measured using a UV-vis spectrophotometer and DAS MA-16 with a glass substrate as a reference. The DAS MA-16 is an instrument that can measure the optical and electrical properties of a thin layer.

The measurement process of optical transmittance from the AgNRs layer was conducted in a closed chamber in the absence of light (dark conditions). A laser diode beam with a wavelength of 630 to 680 nm was fired perpendicular to the surface of the glass substrate without and with coated of AgNRs suspension. The measurement process was performed for 30 seconds for each sample. The optical transmittance rate was obtained by comparing the voltage value of the sample read on the photodiode and AgNRs layer with the blank glass substrate.

The optical transmittance of the AgNRs layer decreased with the number of dipping cycles. The optical transmittance of the AgNRs layer is as shown in Table 1. The optical transmittance of the AgNRs layer with a UV-vis spectrophotometer and DAS MA-16 shows the same value about of 80-90 %. Optical transmittance is an important factor in determining the level of transparency and conductivity of a transparent electrode. The high value of transmittance in a wavelength range of 400-700 nm identifies that the AgNRs layer is excellent to bes used as a transparent electrode of organic solar cells [27, 28].

Sheet resistance (R_s) of the AgNRs layer was measured by using a current-voltage (IV meters) at a voltage range of 0 to 130 mV and a digital multimeter as shown at Figure 4(b). The relationship between the number of dipping cycles, optical transmittance, and a sheet resistance of the AgNRs layer is presented in Table 1. The sheet resistance value is inversely proportional to the number of dipping cycles as shown in Figure 2(d). This condition indicates that the sheet resistance of the AgNRs layer increases with the number of layers. Furthermore, the shape and morphology of the AgNRs solution and the AgNRs layer were observed using SEM at a voltage of 10 kV as shown in Figure 5.

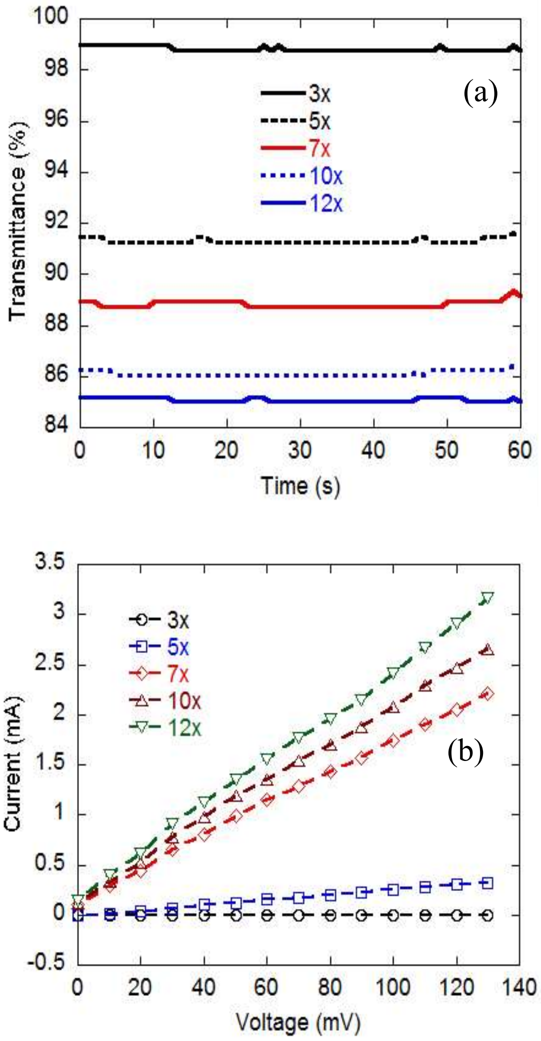

Figure 4. (a)Optical Transmittance of AgNRs Layer Using DAS MA-16 and (b) I-V Meter of AgNRs Layer.

Table 1. Optical Transmittance and Sheet Resistance of AgNRs Layer.

Number of dipping cycles	Optical transmittance (%)	Sheet resistance (Ω sq^{-1})
3	95 ± 3	1500 ± 100
5	93 ± 3	720 ± 50
7	90 ± 3	175 ± 30
10	85 ± 3	90 ± 20
12	80 ± 3	30 ± 5

Figure 5(a) is an SEM image of AgNRs solution which was synthesized using a polyol method. Silver nanorods obtained were with diameter and length average about of 800 µm and 10 to 20 nm, respectively. Figure 5(a) also shows that AgNRs produced uniform and homogeneous without agglomerated with the crystal structure of face-centered cubic (fcc) [23].

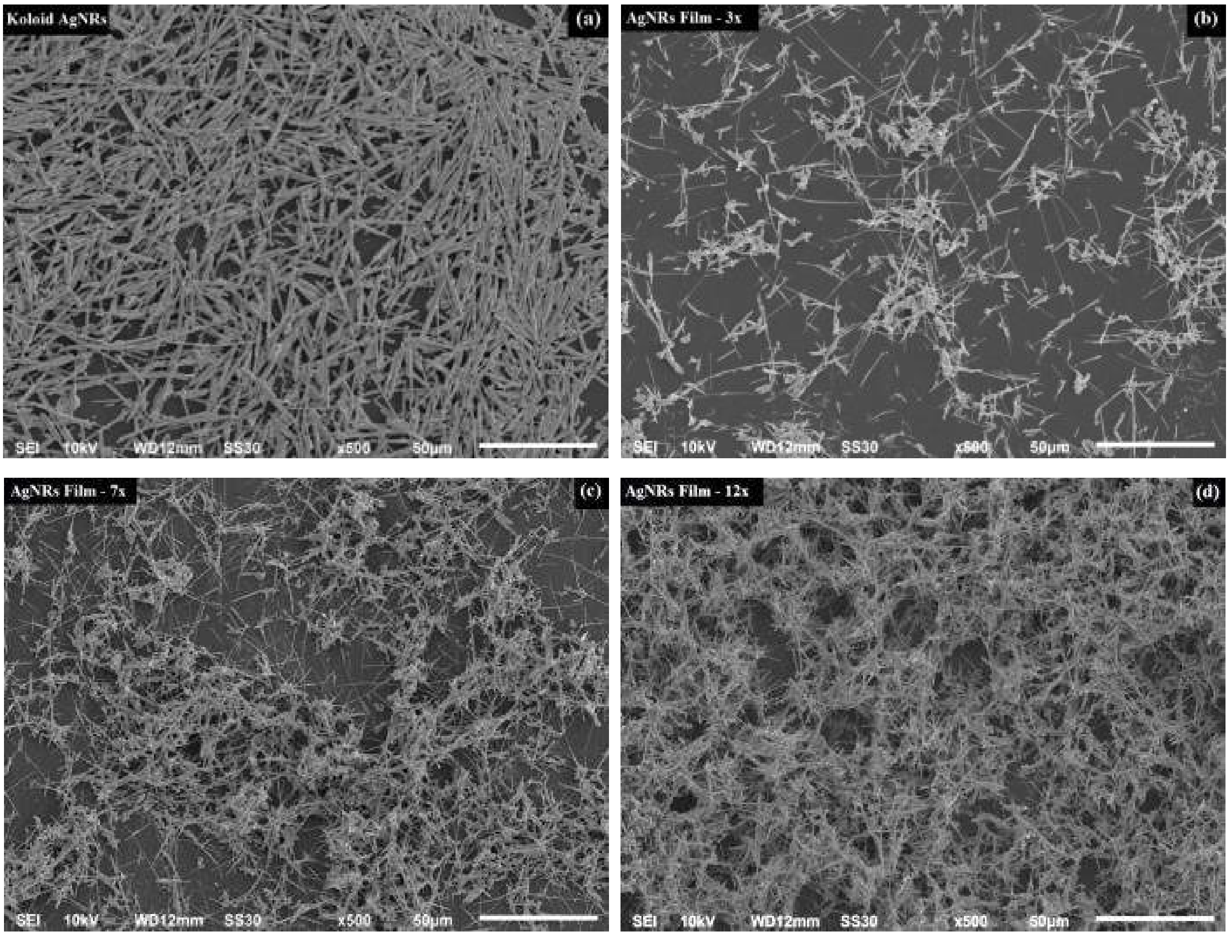

Figure 5. SEM Images of (a) Silver Nanorods Suspension and (b-d) Silver Nanorods Layer.

SEM images for the AgNRs layer with the variation of dipping cyclesare shown in Figure 5(b-c). Figure 5(b) is an SEM image of the AgNRs layer with five times of dipping cycles. According to the SEM image shows that the number of AgNRs deposited onto a glass substrate is slight. This causes the value of the optical transmittance of the AgNRs layer is high. In this condition, the sheet resistance is high, and the electrical conductivity is low. The sheets resistance of the AgNRs layer is very well when the coating process performed for seven times. The number of AgNRs attached to the substrate will increase with the number of dipping cycles. Figures 5(c-d) indicate that repetition of dipping cycles process does not damage or eliminate AgNRs which have first been deposited on the substrate. With the sheet resistance of ~30 Ω sq^{-1} and optical transmittance above of 80%, the AgNRs layer can be used as the alternative of a conductive and transparent electrode for various applications.

IV. CONCLUSION

The synthesis silver nanorods were carried out using ethylene glycol, polyvinyl alcohol, and sodium chloride by controlling the oil bath temperature. The silver nanorods produced in high yield, uniform, and homogeneous without agglomeration occur during the synthesis process. SEM images show the size of the diameter and length of AgNRs of 800 nm and 15 μm, respectively. Optical transmittance of AgNRs layer obtained range from 80 to 90 % with the sheet resistance under of 200 Ω sq^{-1} onto a glass substrate 10 x 25 mm^2. A number of dipping cycles in the dip-coating method are very important for controlling the thickness, optical transmittance, and sheet resistance of the AgNRs layer. The optical transmittance and sheet resistance are linear to the number of dipping cycles. The AgNRs layer is potential as the alternative transparent electrodes of organic solar cells.

ACKNOWLEDGMENT

This work supported by a research grant of "Post Graduate Doctoral, with the contract number 392/UN26.21/PN/2018", by Ministry of Research, Technology and Higher Education (RISTEKDIKTI) of the Republic of Indonesia. Acknowledgments are also extended to Institutes for Research and Community Service, Universitas Lampung.

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