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Thin Films of Silver Nanowires for Flexible, Transparent, and Conductive (FTC) Electrodes

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Abstract*— In this research, we have succeeded in making thin-films for flexible, transparent, and conductive (FTC) electrodes based on silver nanowires. The synthesis of silver nanowires is carried out at low temperatures, namely at 60 to 130 oC. The materials used in the synthesis of AgNWs are polyvinyl pyrrolidone (PVP) as a capping agent and Iron (III) chloride hexahydrate (FeCl3•6H2O) as a precursor to controlling the size of silver nanowires. Furthermore, the silver nanowires colloid then created a thin layer over the polycarbonate (PC) substrate by the roll to roll process. The Result shows that the formation of silver nanowires occurred at low temperatures of about 90 °C. The optimum condition of silver nanowires has obtained synthesis at the temperature of 110 °C with the average diameter of (100 ± 20) nm and length (30 ± 15) µm. The silver nanowires will increase in length and diameter at low-temperature and decrease at high temperatures .The transmittance of FTC film silver nanowires about 76-95% at a wavelength of 550 nm. The absorbance coefficient of FTC film silver nanowires has increased from 2.7 to 29.2 cm-1 at wavelength range 400 to 700 nm. The sheet resistance of the FTC film by varying the number of layers obtained of 905.2, 340.7, 21.9, and 3.4 Ω.sq-1 with the transmittance obtained at 76.7 to 95.8%. The number of layers of silver nanowires will increase the sheet resistance and decrease the optical transmittance of the FTC film.

***Keywords*— silver nanowires; a transparent electrode; flexible substrate; conductive film.**

I. INTRODUCTION

Silver nanowires (AgNWs) attracted many researchers in recent years. Among the advantages of AgNWs include excellent electrical conductivity (6.3×10^{7} $S.cm^{-1}$), high aspect ratio (length up to tenths of micrometers), and fabricated on a flexible substrate. Many applications by AgNWs, such as surface-enhanced Raman spectroscopy (SERS) enhancement [1], [2], transparent conductive electrode [3]–[7], touch screen [8,9], and solar cell [3], [10]. The method for synthesizing AgNWs is a polyol method because of its high yields, low cost, and simple process for growth silver nanostructures. Fievet and group demonstrated the first polyol method for synthesizing colloidal metal nanoparticles. Afterward, Sun and the group applied this method for synthesizing AgNWs [11]–[15]. The temperature is a crucial parameter in the synthesis of AgNWs. The lowest temperature that can use for the synthesis AgNWs was 110 °C [16]–[18].

Many papers have published on the synthesis of AgNWs at high temperatures (above 100 °C). A thin layer of AgNWs deposited on a substrate such as polyethylene terephthalate (PET) almost entirely writes that this material has high transmittance and low resistance. However, studies of the optical and electrical properties of AgNWs films have not been published yet.

Transparent conductive films (TCFs) has been reported by Coleman et al. comparable to the parameters of ITO thin films [19]. The AgNWs thin films revealed an optical transmittance and sheet resistance of 85% and 13 $\Omega.sq^{-1}$, while the ITO thin films of 90% and 10 $\Omega.sq^{-1}$. Furthermore, TCFs based on AgNWs can be used in several kinds of optoelectronic devices [21,22]. Although the performance of the electrical and optical AgNWs thin-film looks promising, the production of homogeneous, less surface roughness, and the application for organic devices still needs more investigation.

The TCFs based on AgNWs were produced by several techniques such as dip coating, Meyer rod coating, drop-casting, brush painting, spray coating, and spin coating. In this work, we demonstrate low temperatures in the typical polyol process for synthesizing AgNWs. This work aimed to acquire the possibility of silver nanowires growth and estimate reaction time at low temperatures. The synthesis of AgNWs is carried out with temperature variations from 60 to 130 ^{o}C. Therefore, the Meyer rod coating or roll-coating technique is widely used to fabricate FTC film silver nanowires using a polycarbonate substrate. The study of optical and electrical properties are performed to determine optical transmittance, sheet resistance, refractive index, bandgap energy, and optical conductivity of FTC film silver nanowires.

II. Materials and Methods

A. Materials

The materials were used for synthesis and fabrication FTC film of AgNWs included: silver nitrate ($AgNO_3$, Sigma-Aldrich), ethylene glycol (EG, 99%, Merck), deionized water, polyvinyl pyrrolidone (PVP, MW = 55000 g/mol, Sigma-Aldrich), Iron (III) chloride hexahydrate ($FeCl_3$•6H_2O, Merck Millipore), ethanol (EtOH, 98%, Merck), and polycarbonate (PC) substrate.

B. Method

1) Synthesis of silver nanowires (AgNWs): PVP of 0.399 g was mixed in 8 mL ethylene glycol at 350 rpm for five minutes. Furthermore, $FeCl_3$•6H_2O/EG solution 0.1 M as much 10 μL was added rapidly into PVP/EG solution and the mixture pre-heated at 90 ^{o}C for 50 minutes. In the next step, 4 ml $AgNO_3$/EG (0.3 M) solution injected into the mixture after the temperature was stable. The PVP, $FeCl_3$•6H_2O, and $AgNO_3$ in EG solution continue heated and stirred until was obtained from AgNWs solution. The reaction was stopped when the silver nanowires at the optimum condition.

2) Electrode Fabrication: Fabrication of FTC film silver nanowires was done on a polycarbonate substrate of 15×20 cm^2 by varying the number of layers using a bar-coater (RDS-20). Firstly, the PC substrates ultrasonicated in ethanol at room temperature and dried at ambient conditions. The silver nanowires colloid was dispersed in an ethanol solution with a concentration of 10 wt.%. Finally, the FTC film silver nanowires were dried on an oven at 90 ^{o}C for 15 min.

C. Characterization

The silver nanowires colloid was characterized by using UV-vis spectroscopy, FTIR spectroscopy, and scanning electron microscopy (SEM, JSM-6510). The four-point-probe (Keithley) were used to measure the electrical properties of FTC films by varying the electric current from 0 to 0.5 mA. For measuring the optical properties of the FTC film silver nanowires, were used UV-vis spectroscopy in the wavelength of 300 to 800 nm. The morphology, size, and thickness of FTC film silver nanowires were observed using SEM.

III. Results and Discussion

The characterization of the optical properties of the silver nanowires solution using UV-vis spectroscopy obtained absorption spectra for several forms, such as nanoparticles, nanorods, and nanowires. The color of the resulting solution will differ depending on the catchment area of the ultraviolet (UV) and visible (vis) at specific wavelengths. For silver nanoparticles that absorb at the blue and violet wavelengths around 400 to 510 nm [21]–[23]. In order to the silver nanowires surrounding areas that absorb at the UV-vis spectra. Optical properties of silver nanowires colloid and FTC film silver nanowires as shown in Fig. 1.

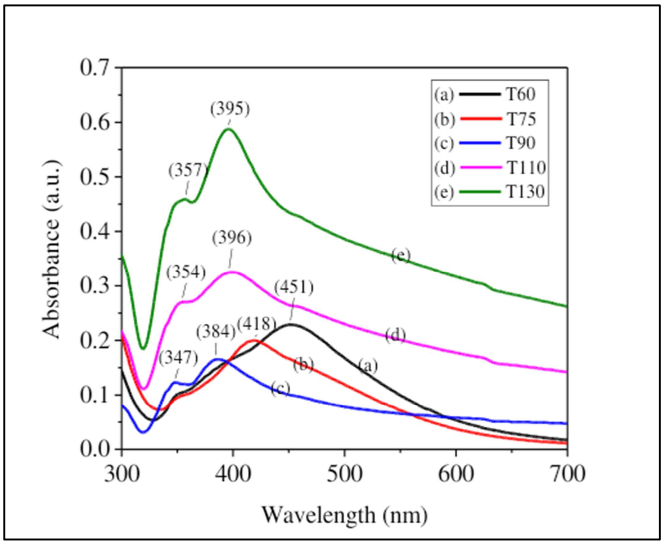

Fig. 1 UV-Vis spectra of AgNWs colloid with different temperature

Fig. 1 shows the UV-vis spectra of silver nanowires at a temperature of 60 ^{o}C obtained a peak at 418 nm. Based on the literature, the absorption peak is silver nanoparticles while the broad peak was indicating the large distribution in size [14]. This result strengthened by the SEM images of the product (Fig. 5a), which shows the structures of the silver nanowires with widespread distribution in diameters. It observed that the peak at 458 nm at the temperature of 75 ^{o}C, identifying the final product was silver nanowires. Two peaks were appeared representing the absorbance of silver nanowires synthesized at a temperature of 90, 110, and 130 ^{o}C. The first peak located at the ~380 nm and the second peak located at ~350 nm. In our case, the first peak for sample 90, 110, and 130 ^{o}C located at the wavelength of 395 nm, 396 nm, and 384 nm, respectively. These peaks associated with transverse mode of surface plasmon resonance of silver nanowires. The other peak describes to quadrupole plasmon resonance located at the wavelength of 357 nm, 354 nm, and 347 nm. The optical transmittance of FTC films based on AgNWs showed in Fig. 2.

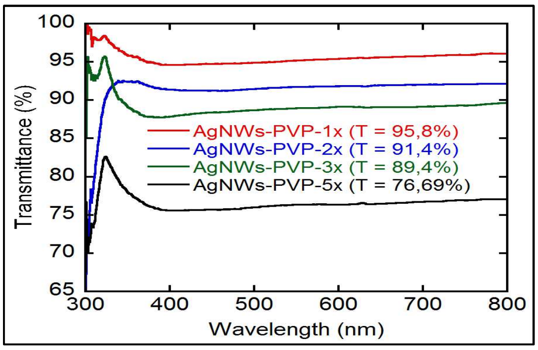

Fig. 2 UV-Vis spectra of transmittance of FTC film silver nanowires.

In Fig. 2, the transmittance of FTC film silver nanowires about 76-95%. The optical transmittance of the FTC film is stable at a wavelength of 550 nm at the wavelength of visible light. The thin result shows that FTC film silver nanowires can be used for transparent conductive electrodes (TCEs). Fig. 3 shown FTIR spectra of PVP powder and the silver nanowires synthesized at a temperature of 90 ^{o}C, 110 ^{o}C, and 130 ^{o}C.

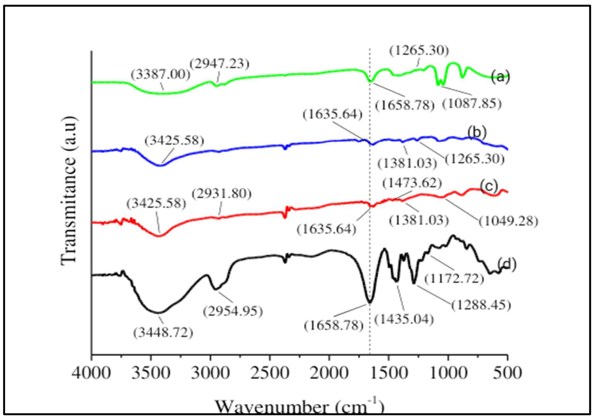

Fig. 3. FTIR spectra of different sample AgNWs synthesized at: (a) 130 ^{o}C, (b) 110 ^{o}C, (c) 90 ^{o}C, and (d) PVP powder.

FTIR characterization was performed to acquire information about surface capping of PVP on the formation of silver nanowires. To ensure no PVP residual on the silver nanowires surface, FT-IR spectra can detect the surface that capped by PVP for all samples. The synthesis of silver nanowires in a variation of temperature formed O-H stretching vibration located at 3425.58 cm^{-1}, 3425.58 cm^{-1}, and 3387 cm^{-1}. The peaks located at 2931.80 cm^{-1} at 90 ^{o}C and 2924.09 cm^{-1} at 110 ^{o}C are indicating –CH_2– stretching vibration [22]. The weak peak located at 2947.23 cm^{-1} at 130 ^{o}C can be assigned to –CH_3 stretching. For AgNWs at 90, 110, and 130 ^{o}C are located at 1635.64 cm^{-1}, 1635.64 cm^{-1}, and 1658.78 cm^{-1} are indicates the C=O stretching vibration. For PVP powder (Fig. 3d), a broad peak at 3348.72 cm^{-1} is ascribed to O-H stretching vibration. The absorption peak around of 2954.95 cm^{-1} and 1658.78 cm^{-1} indicates the stretching vibration of C-H and C=O.

According to this result, carbonyl absorption red-shifted from 1658.78 cm^{-1} to 1635.64 cm^{-1} at 90 and 110 ^{o}C. This probably occurs because the oxygen of the carbonyl gets a pair with the Ag atom. Even the centrifugation has been carried out; the PVP residual shows at 130 ^{o}C. The peak for carbonyl has the same location at 1658.78 cm^{-1} with PVP. This capping will reduce the conductivity degree of nanowires [24,25]. The diameter has slightly changed at 90 °C and 110 °C which shown in Fig. 3. SEM images of silver nanowires have been synthesized with various temperature as shown in Fig. 5.

SEM images in Fig. 4a at the temperature at 60 °C show that only produced the silver nanoparticles with a size of about 86 nm. In this condition, oil bath temperature is too low have not been able to break the atomic bonds of oxygen (O) atom form ring of pyrrolidone (-NO-) owned by PVP and no interaction between Ag atom by O atom through the Ag-O bond in the formation of silver nanowires [25].

The silver nanowires homogeneously formed when synthesized at a temperature above 75 °C. Fig. 4 and Fig. 5 show that silver nanowires are well-formed when synthesized at a temperature of 90 to 130 °C.

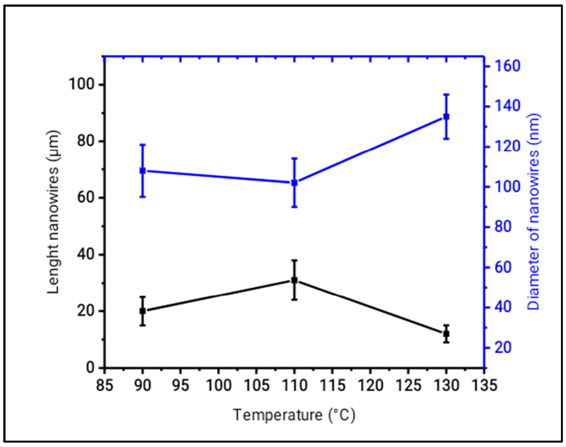

Fig. 4. Lengths and diameters of AgNWs as a function of time.

The synthesis of silver nanowires with an oil bath temperature of 90 °C to produce silver nanowires with diameter and length about of (108 ± 25) nm (20 ± 10) μm, respectively (Fig. 5b). The diameter and length of silver nanowires gained about of (102 ± 24) nm (31 ± 15) μm when synthesized at a temperature of 110 °C as in shown Fig. 4c. The synthesis of silver nanowires at a temperature of 130 °C (Fig. 5d), shows the increase of the diameter of silver nanowires around of (135 ± 22) nm and the decrease of length about (12 ± 6) μm.

The silver nanowires successfully synthesized at 130 °C. The products have appeared in about 30 minutes. Later, the reaction stopped after 60 minutes. In this case, higher temperature produces more reducing agent which influence to shorter reaction time. Lowering temperature from 130 °C to 110 °C takes the longest time. An optical microscope recorded the growth of silver nanowires at 110 °C. The silver nanoparticles formed in about 110 minutes of reaction time, after 150 minutes, the rods like structure appeared, and its increase in length grew into nanowires after 220 minutes. Anisotropic growth continued until 240 minutes. The heating temperature of 90 °C takes 22 hours to obtain the silver nanowires due to the slow reduction process. This result was different from Sun et al. work. They reported no nanowires formed after mixture heated at 100 °C for 20 hours. Heating under 90 °C can't produce nanowires, and it has only formed nanoparticles. The reaction finished in 52 hours and 72 hours, at the 75 °C and 60 °C, respectively.

The thermal condition at 110 °C was rapidly reducing precursor and serve enough Ag nuclei on the solution. It becomes possible to improve in NWs length. In contrast, Excessive thermal energy could cause the seed growing into short wires, which can find at 130 °C. The large difference found at 130 °C. It is still unclear why a significant increment occurs in that thermal environment. Widely known, that the silver nanowires will increase in length and diameter at low-temperature and decrease at high temperatures [14]. Willey et al. investigated the effect of Fe^{3+}. EG forming Fe^{2+} reduces the Fe^{3+} ion, then Fe^{2+} absorbs oxygen, which causes the reduction of oxygen in the reaction [26]. Our data shows that Fe^{3+} promotes the growth of wires [27].

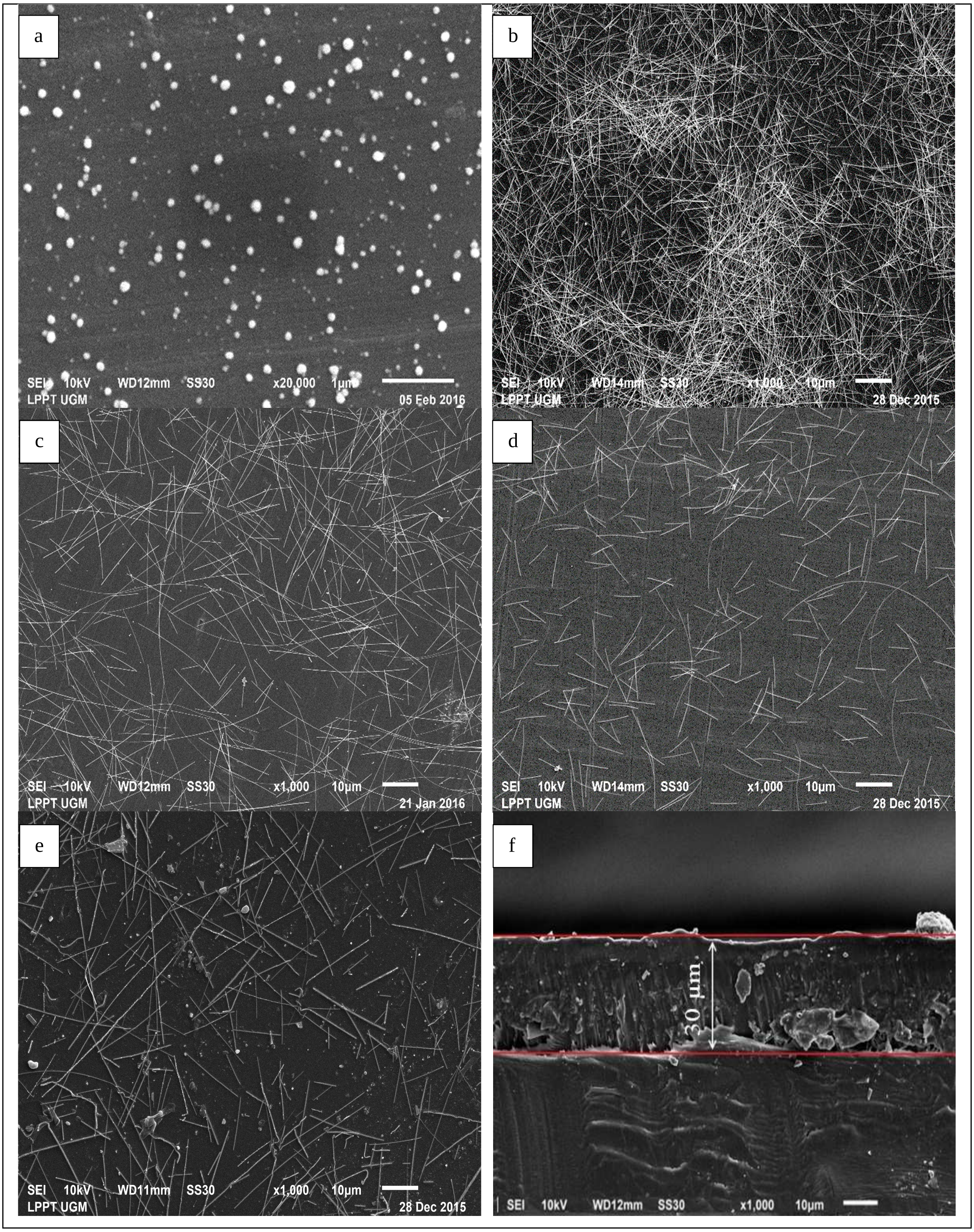

Fig. 5. SEM images of silver nanowires at temperature of (a) 60 °C, (b) 90 °C, (c) 110 °C, and (d) 130 °C (e-f) FTC film silver nanowires.

The silver nanowires used for the manufacture of thin films are the result of synthesis at 90 °C (Fig. 5b). The silver nanowires deposited on a polycarbonate substrate with a Meyer-rod coating technique are evenly distributed as shown in Fig. 5e. The thickness of the silver nanowires thin layer was observed using SEM by cross-section technique. The thickness of the silver nanowires thin film is obtained about 30 µm as shown in Fig. 5f.

Absorption spectrophotometry, such as UV-vis and IR spectroscopy, is the measurement of an interaction between electromagnetic radiation and the molecule or atom of a chemical. The molecule always absorbs electromagnetic radiation if the radiation frequency is equal to the vibrational frequency of the molecule. Both bound and unbound electrons will be excited at a frequency region, which corresponds to UV-vis radiation. The wavelength of the UV spectrum region is 190-380 nm, while the visible spectrum is 380-780 nm. The corresponding spectrophotometer for measurement in the UV-vis spectral region consists of an optical system with the ability to produce monochromatic light within the range of 200-800 nm and an appropriate means for determining absorption.

To determine the optical properties of the FTC film silver nanowires, transparency tests were performed with a UV-vis spectrometer at 300 to 800 nm wavelength. The UV-vis spectrometer used to measure the absorbance (A) and transmittance (T) of the FTC film samples. The absorbance is a light polarization that is absorbed by a chemical material at a certain wavelength so that it will give a specific color to the material. While the transmittance is the fraction between the intensity of incoming radiation (I_0) to the exit intensity (I) of the material with a certain thickness. From these two values, we can determine the values of reflectance (R), absorption coefficient (α), refractive index (n), energy gap (E_g), and optical conductivity (σ) of silver nanowires thin layers. Calculation of these parameter values can be done using the following equation:

$$\alpha = 2.303\frac{A}{d} \quad (1)$$

$$A + T + R = 1 \quad (2)$$

with d is the thickness of the thin layer of silver nanowires. Using the absorbance coefficient value obtained at Eq. (1), we can calculate the energy gap value of the silver nanowires thin layer. Energy gap calculation is done by using Eq. (3).

$$(\alpha h\upsilon) = B\left(h\upsilon - E_g\right)^{\gamma} \quad (3)$$

with ν = the frequency of the light wave (Hz) equal to the value (c / λ). Where $\boldsymbol{B}$ is a probable transition factor value, and γ is an index value that depends on the type of electronic transition. For a thin layer of silver nanowires, the value γ = ½ when a direct transition occurs, and γ = 2 when a direct transition occurs. From the absorbance and reflectance coefficient values, we can determine the refractive index value, the extension coefficient (k), and the optical conductivity (σ) of the silver nanowires thin layer sample using Eq. (4), (5) and (6). The electrical conductivity is a measure of the ability of a material to conduct an electric current.

$$n = \left(\frac{1+R}{1-R}\right) + \left(\frac{4R}{(1-R)^2} - k^2\right)^{\frac{1}{2}} \quad (4)$$

$$k = \frac{\alpha\lambda}{4\pi} \quad (5)$$

$$\sigma = \frac{\alpha n c}{4\pi} \quad (6)$$

with λ is the wavelength (m) and $\boldsymbol{c}$ is the speed of light (3×10^8 m.s^{-1}) [31,32]. When there is an excited electron from the valence band to the conduction band, its minimum energy will be equal to its gap energy. So, the gap energy can also be defined as the minimum energy required by the electrons in the valence band to move toward the conduction band. Between the valence band and the conduction band, there is a gap where the electrons will jump from one band to the other. This gap will show the properties of a solid, whether the solid is a conductor, an insulator, or a semiconductor. The energy gap graph of the silver nanowires thin layer as shown in Fig. 6.

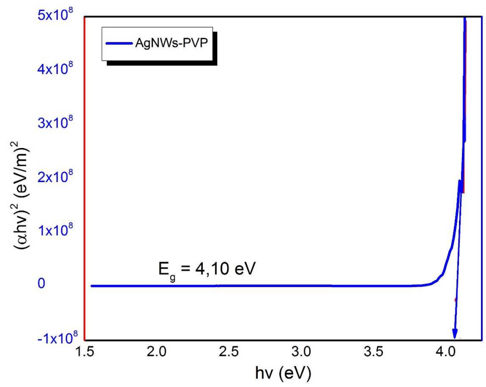

Fig. 6. The energy gap of FTC Films silver nanowires.

From the calculation result using Eq. (3), the energy gap for the silver nanowires thin layer is 3.6 eV. The energy gap generated from this study corresponds to the energy gap of the previous silver nanowires' thin layer results. According to the literature, the energy gap for thin-film ZnO-AgNRs-PVP is about 3.66 to 3.71 eV [32]. The energy gap value of Ag metal is about 3.1 to 5.2 eV [21], [34]. The energy gap is highly dependent on the value of the absorbance coefficient on photon energy. Fig. 7 shown the FTC film silver nanowires.

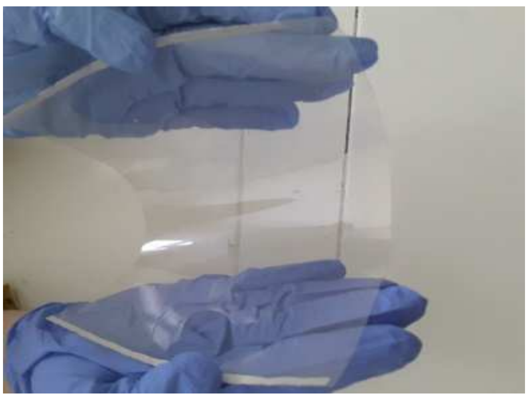

Fig. 7. The FTC films coated on a flexible substrate.

Fig. 6 shows an FTC film silver nanowire coated in silver nanowires solution with a roll to roll technique on a 15x20 cm^2 polycarbonate substrate. The coating process is performed per layer by varying the number of layers. Every single coat, the FTC film were dried on an oven at 90 ^oC for 15 min. The absorbance coefficient of FTC film silver nanowires has increased from 2.7 to 29.2 cm^{-1} at wavelength range 400 to 700 nm. The increase of absorbance coefficient value is accompanied by increasing of photon energy from silver nanowires thin film that is equal to 1.8 to 3.1 eV [35]. The value of the absorbance coefficient can also be calculated as the value of the extension coefficient of silver nanowires thin film. The value of the extension coefficient decreases with the increased wavelength exposed to the sample [36].

From the absorbance coefficient value and refractive index, the optical conductivity will be generated as in Eq. (6). In Fig. 8, the optical conductivity value for the FTC film silver nanowires coating is obtained about 13.1×10^5 $S.m^{-1}$. The optical conductivity of the FTC film silver nanowires is close to the optical conductivity value of a thin layer of silver nanowires coated on the substrate polydimethylsiloxane substrate (PDMS). According to a study, the optical conductivity of the silver nanowires thin layer sample deposited on the PDMS is about 8.13×10^5 $S.m^{-1}$ [38]–[41].

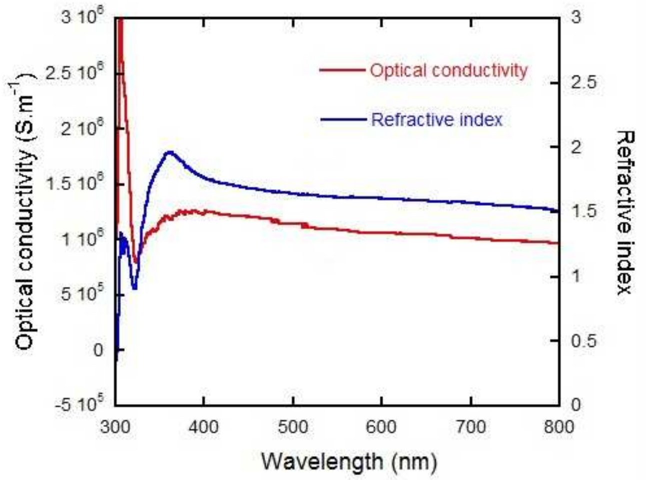

Fig. 8. Optical conductivity and refractive index of FTC film silver nanowires.

The refractive index is calculated by using Eq. (4). Fig. 8 shows that the refractive index decreases with the rise of the wavelength. The refractive index value is saturated at a wavelength of about 450 nm, which is 1.7. When the wave through two different mediums, it will experience the event of refraction. The refraction of light is a reversing event of light velocity when entering one medium to another medium. The magnitude of the deflection or the shifting of the light creeping direction coming out of a medium depends on the optical density of the medium. This optical density is the nature of a translucent medium (optical agent) in passing light. The refractive index of the medium is defined as the ratio of the velocity of light in a vacuum to the speed of light in a medium. The larger the refractive index of a substance the greater the light is deflected by the substance. The magnitude of refraction also depends on the wavelength of light. In the visible light spectrum, the wavelength of light varies from the longest red wave to the shortest UV wave. [41].

The amount of light on the surface of the substrate will decrease the extension coefficient of the FTC film silver nanowires. This condition is caused by reduced light fraction due to decreasing light intensity caused by the sample attached to thin film [36]. The sheet resistance curve of FTC film silver nanowires as shown by Fig. 9.

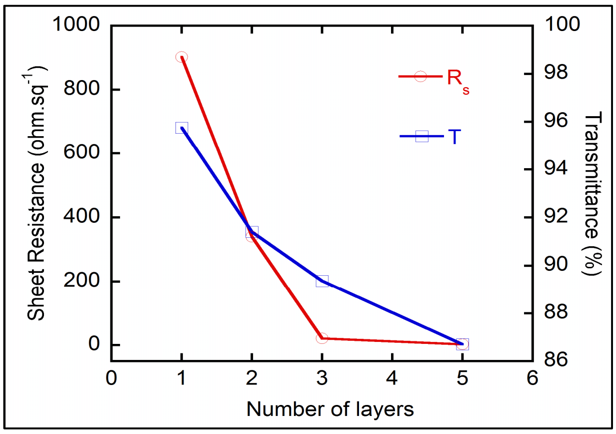

Fig. 9. Sheet resistance and transmittance of FTC film silver nanowires.

Fig. 9 shows that the sheet resistance of the FTC film by varying the number of layers obtained of 905.2, 340.7, 21.9, and 3.4 $\Omega.sq^{-1}$, respectively. When the sheet resistance decreases, the optical conductivity of the FTC film silver nanowires will increase. The transmittance of the FTC film is obtained at 76.7 to 95.8%. Increasing the number of layers will decrease the transmittance of the FTC film silver nanowires. The energy gap was calculated using the absorbance value of the UV-vis measurements of the FTC films. Uniformity and non-homogeneity of the thin film will cause a decrease in optical and electrical properties of the silver nanowire's thin layer [22], [42].

IV. Conclusions

Temperature is the critical parameter on the synthesis of silver nanowires. In this result, silver nanowires grow optimally when synthesized at a temperature of about 90 to 130 ^oC. The lower the temperature, the synthesis of silver nanowires will take place more slowly. The optical and electrical properties of the FTC film were critically dependent on the number of layers of silver nanowires solution. The thickness of the FTC film is obtained about 30 μm by the sheet resistance and the optical conductivity of 3.4 $\Omega.sq^{-1}$ and 4.7×10^5 to 13.1×10^5 $S.m^{-1}$, respectively. Next, the refractive index of FTC film is about 1.2 to 1.7. In the manufacture of thin layers, the main problem faced is the difficulty of obtaining a uniform and homogeneous layer. This condition is caused by many factors, such as pressure press, the speed of the coating process, and the temperature to dry the coating after the coating.

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