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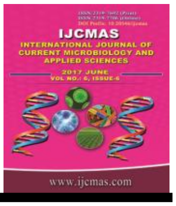

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Electrical Conductivities, Photocurrent Densities and Mechanical properties of Ag/TiO_2 Composite Thin Films Heat Treated at Different Temperatures

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ABSTRACT

The control of heat treatment temperature plays a key role towards understanding the effect of different temperatures on the optical, photo responsive, electrical conductivity and mechanical properties of noble metal/semiconductor composite thin films. This work reports on the electrical conductivity, photo electrochemical, and mechanical properties of silver nanoparticles/titanium dioxide (Ag-NP/TiO_2) composite thin films, successfully synthesized. Respective precursor solutions for Ag-nanoparticles and titania were prepared from Ag salt and a titanium complex using the molecular precursor method (MPM), spin-coated on quartz glass substrates and heat treated at different temperatures, namely; 250, 300, 400, 500, 600, 700 and 800 °C. The electrical resistivity of the films was of the order of 9.6×10^{00} to 10^{-4} $\Omega\cdot$cm with film thicknesses in the range 100–270 nm. For the study of electrochemical properties of the Ag-NP/TiO_2 composite thin films, the photocurrent was measured under natural potential by a conventional three-electrode method using a silver (Ag) plate as a counter electrode. Under Vis-light irradiation, the cathodic photocurrent density of these films increased with an increase in the heat treatment temperature. The mechanical strength of quartz glass, and Ag-NP/TiO_2 thin films whose heat treated at higher temperature (500, 600, and 700ºC) were examined in order to check their potential use in practical systems as solar cells. The results illustrated that there is a distinctive decrease of Young`s modulus and Knoop hardness values with an increasing of heat treatment temperature.

Keywords

Electrical conductivities, Photocurrent densities, Temperatures.

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Introduction

In recent times, extensive studies have been focused on searching the best materials for multifunctional materials thin film. The main advantages for these materials are low cost, environmental friendly, high efficiency and stability. TiO_2 has been a strong candidate due to its high stability in aqueous solutions (Nishide *et al*., 2000) and high photovoltaic (Liu *et al*., 2010) and photo catalytic activity (Wang *et al*., 2013; Daniel *et al*., 2013). Nanotechnology, which manipulates materials at the nano or atomic scale, has a great potential for design and synthesis of multifunctional materials with desired and unique properties. It can also reduce the cost of materials manufacture. The objective of the work is to pursue the possibility of using Ag/TiO_2 composite films to understand their optical, photo responsive, electrical conductivity and mechanical properties. It is well-known that the changes in the profiles of absorption spectra at different heat treatment temperatures can be attributed to the changes in the surface morphology of the thin films (Hasan *et al.*, 2008). This can be characterized by studying absorbance using UV-visible spectroscopy. Moreover, a systematic study of size effect on the electrical properties of semiconducting nanocrystallites is essential for understanding their technological applications (Sarah *et al.*, 2010). Electrical conductivity is the ability of a material to conduct an electric current, when there is an electrical potential difference placed across a conductor, its movable charges flow, giving rise to an electric current (Wang *et al.*, 2012).

The enhanced near-field amplitude of surface plasmon resonance (SPR) in the proximity of metal nanoparticles can boost the photoactivity of the neighboring semiconductor, which has been proven and has attracted wide interest recently. One best way to prove this, is simply by studying the photo electrochemical properties of the doped semiconductor. Most studies on the photo electrochemical mechanism of noble metal/TiO_2 composites have been focused on the details of photo induced electron transfer, which can be used to determine the vis-light response of the composites, though the Ag content in these composites is quite low (3−6 mol%) (Zhao *et al.*, 1997; Li *et al.*, 2008).

Nanocrystalline metallic composite thin films show potential for engineering application due to their high strength[10]. In this paper, firstly the electrical conductivities properties of Ag-NP doped in titania matrix will be investigated. Finally, the photo electrochemical properties and the mechanically strength of Ag-NP/TiO_2 thin films whose heat treated at higher temperature (500, 600, and 700ºC) will be examined in order to check their potential use in practical systems.

Materials and Methods

Fabrication process of Ag/TiO_2 composite thin films by coating and heat treatment

The precursor solution containing the Ti^{4+} complex of EDTA was obtained by a molecular precursor method that we previously reported (Daniel *et al.*, 2013; 2012). Dibutylamine (3.58 g, 27.7 mmol) and EDTA (3.56 g, 12.2 mmol) were added to a mixture of 10 g of ethanol and methanol. The solution was refluxed for 2 h with stirring and then cooled to room temperature. After adding 3.47 g (12.2 mmol) of $Ti(O^iPr)_4$, the solution was refluxed for 4.5 h. The reaction mixture was cooled to room temperature, and 1.56 g (13.8 mmol) of 30% H_2O_2 was carefully added. The solution was then refluxed for 0.5 h. The concentration of titanium was 0.4 mmol g^{-1}.

The Ag acetate ethanol solution for the preparation of Ag-nanoparticle fabrication in the composite thin films was prepared according to the method we reported recently (Daniel *et al.*, 2015). Silver acetate (0.24 g, 1.4 mmol) and dibutylamine (0.56 g, 4.3 mmol) were added to 1 g of ethanol. The solution was sonicated with stirring for 5 min. The concentration of silver was 0.8 mmol g^{-1}.

The two prepared precursor solutions were then mixed at 1:1 molar concentration to form composite solution as a thin film coating precursor solution. The coating precursor solutions were deposited by spin coating onto the cleaned quartz glass substrates with a double step mode: first at 500 rpm—5 s and then at 2000 rpm—30 s in all the cases. The resultant composite thin films were fabricated by heat-treatment at different temperatures (250–800ºC) for 0.5h. Thin films of pure TiO_2 were also fabricated for comparison purpose by heat treating the spin-coated S_{Ti} precursor films at 800ºC.

Absorption spectra of the resultant thin films

The absorption spectra for the TiO_2, Ag NP, and COMP-Ag*n* thin films fabricated on quartz glass substrates were measured in the range 200–800 nm using the double-beam mode. The measurement was performed using a spectrophotometer (U-2800, Hitachi) and air was used as a reference.

Measurement of the film thickness and the electrical resistance of the resultant thin films

The film thicknesses of titania and COMP-Ag*n* films were measured using a stylus profilometer (DEKTAK3, Sloan). The electrical resistance at 25°C was measured using the four probe method involving two multimeters (VOAC7512, Iwatsu and Model 2010 Multimeter, Keithley) and a regulated DC power supply (Model PAB 32-1.2, Kikusui Electronics Corp.). Four gold-plated tungsten probes (FELL type, K&S) were placed at intervals of 1 mm, and an added load of 0.2 kg was applied. The electrical resistivity, ρ, of the films was calculated using equation (1):

$$\rho = cRt \qquad (1)$$

Where,

c, R, and t represent the correction value (4.45), electrical resistance, and film thickness, respectively.

Photo electrochemical properties of the resultant thin films

The conventional three-electrode system[11] was employed for photocurrent density measurements. TiO_2 and Ag-NP/TiO_2 composite thin films with different heat treatment were used as the working electrode. A Cu wire was attached to the sample surface by a carbon foil.

In all cases, an Ag metal plate, with a size equal to that of the working electrode, and a Ag/AgCl electrode were used as the counter and reference electrodes, respectively. The photocurrent density of the sample electrodes was measured under Xe light irradiation (passed through spectra filters) from a lamp (Lax-Cute); under UV light irradiation in the wavelength range 300–400 nm from Lax-UV(300–400); and under Vis-light in the wavelength range 400–700 nm from Lax-Vis(400–700). The light intensity for the latter two irradiations was 8.0×10^4 lux. All the measurements were performed in a 0.1 mol/L Na_2SO_4 solution after bubbling Ar gas at 50 mL/min for 10 min. The flow rate for the Ar gas bubbling was equal for all measurements.

The photocurrent densities were obtained at 10 s intervals by irradiating the samples with the chosen light for 30 min. The photocurrent density was recorded using a Toho Technical Research galvanostat/potentiostat (model: 2090) under natural potential. The average photocurrent density (APD) was calculated from three independent measurements using different films, according to the equation (2):

$$APD = \frac{\sum PD}{\text{No. of data}} \qquad (2)$$

Where,

PD is the photocurrent density measured every 10 s for 30 min after switching on the light. Dark current—current without light irradiation—was also recorded during the measurements and the average dark current density (ADD) was calculated according to the equation (3):

$$ADD = \frac{\sum DD}{\text{No. of data}} \qquad (3)$$

Where,

DD is the current density measured every 10 s for 30 min between switching off and on the light.

Mechanical properties of the resultant thin films

The indentation, scratch, and imaging techniques were employed for evaluation of mechanical and tribological properties of the fabricated thin film specimens using a scratch test of the coated film was performed using a scratch tester (HEIDON-22, Shinto Scientific) equipped with a Rockwell diamond C stylus of 200 Am radius and friction force measurement. The measurements were made at progressive loads from 0 to 30 N. The stage speed was 0.40 mm/s and the stylus was pressed on a sample at the rate of 0.26 mm/s. All scratch traces were observed by laser microscope. Its software allows for image analysis to obtain scratch depth profile along any direction of the image. The hardness values were obtained from equation (4):

$$HS = \mathrm{H}_R \times \left[\frac{f_s{}^2}{f_R}\right] \times \left[\frac{W_R^2}{W_S^2}\right] (4)$$

Where,

HR is the hardness of the reference material (quartz glass in this case), WR and WS are the scratch widths on the reference and specimen, respectively.

The FS and FR are the scratching loads for the specimen and the reference, respectively.

Young`s modulus has been calculated from an equation (5), which is dominated by the accuracy of the bar`s width (w) and length (l), but especially by its thickness (t):

$$E = 0.9465 \times \left[\frac{m \times f_r{}^2}{b}\right] \times \left[\frac{L^3}{t^3}\right] (5)$$

Three samples were selected for each condition and tests were performed twice for each sample.

Results and Discussion

The precursor solution, for fabricating silver films could be obtained by dissolving an appropriate amount of silver acetate in ethanol in the presence of dibutylamine. Furthermore, the titania precursor solution, involving the Ti–EDTA complex was also successfully prepared. Seven composite precursor solutions with 1:1 molar percentages of Ag to Ti-EDTA, could be facilely obtained by mixing the two solutions. The composite thin film were heat treated at different temperatures namely; 250, 300, 400, 500, 600, 700 and 800 °C to obtain seven (7) Ag/TiO_2 composite thin films.

Optical properties of the Ag/TiO_2 composite thin films

Figure 1 (a) and (b) represent the UV-Vis absorption spectra for Ag/TiO_2 composites thin films heat treated at different temperatures. The absorbance spectra of the synthesized Ag-NP/TiO_2 composite thin films are decreasing, and then increasing in the visible region with heat treatment temperatures. Composite thin film heat treated at 600ºC exhibit the highest absorption spectrum and well define SPR signatures compare to other higher temperature heat treated composite thin films as shown in figure 1(b).

Furthermore, an absorption band at 395 nm starts appearing in absorption spectra of the prepared Ag-NP/TiO_2 composite films heat treated at temperature greater than or equal to 500ºC, shows clearly that the presence of large amounts of silver on the surface of the thin films due to agglomeration facilitates SPR, which shifts to shorter wavelength with increase in heat treatment temperature.

Composite thin film heat treated at 600ºC exhibit the highest absorption spectrum and well define SPR/LSPR signatures compare to other higher temperature heat treated composite thin films as shown in figure 2.

Electrical resistivity of Ag/TiO_2 composite thin films heat treated at different temperatures

A systematic study of size effect on the electrical properties of semiconducting nanocrystallites is essential for understanding their technological applications (Chellammal *et al.*, 2010). Electrical conductivity is the ability of a material to conduct an electric current, when there is an electrical potential difference placed across a conductor, its movable charges flow, giving rise to an electric current (Oldham *et al.*, 2012). Figure 2 shows the impedance spectrum for various temperature regions of prepared Ag/TiO_2 composite thin film samples obtained used a 4-point probe measuring system.

At low temperature region, that is 70–400ºC, the resistivity varies from 9.6×10^{00} to 1.7×10^{00} Ω cm. As the temperature is further increased, from 400 to 600ºC, the electrical resistivity values of thin films are found to be decreased from 1.7×10^{00} to 1.1×10^{-4} Ω cm. At the higher temperature region, 800ºC, the electrical resistivity values have decreased considerably, from 1.1×10^{-4} Ω cm to undetectable conductivity (out-layer).

Photo electrochemical property of Ag/TiO_2 composite thin films heat treated at different temperatures

Anodic photocurrent could be observed in the composite thin films under dark, visible and UV-light irradiation as shown in figure 3 and the average values of current (APD and ADD) are tabulated in table 1.

Dark cathodic currents were observed in all composite thin films heat treated at different temperature. The cathodic photocurrent for Ag/TiO_2 thin film under UV-irradiation is very low compare to cathodic photocurrent experienced under visible light. Under vis-light irradiation, the cathodic photocurrent density of these films increased with an increase in the heat treatment temperature.

Mechanical strength of the thin films

The mechanically strength of quartz glass, and Ag-NP/TiO_2 thin films whose heat treated at higher temperature (500, 600, and 700ºC) were examined in order to check their potential use in practical systems. The mechanical strength of pure TiO_2 fabricated at 600ºC was also investigated for comparison purpose. Figure 4 shows load-displacement curves obtained from indentation tests on the composite thin films.

Table 2 shows the Young`s modulus and Knoop hardness results obtained during the measurement of mechanical strength of the samples. The results illustrated that there is a distinct decrease of Young`s modulus values with an increasing of heat treatment temperature.

Figure 1 (a) and (b) represent the UV-Vis absorption spectra for Ag/TiO_2 composites thin films heat treated at different temperatures. As shown in these figures, the absorbance spectra of the synthesized Ag-NP/TiO_2 composite thin films are decreasing, and then increasing in the visible region with heat treatment temperatures, indicating that the heat treatment temperatures are capable of sensitizing Ag/TiO_2 thin film. For Ag/TiO_2 composite thin film heat treated at 70ºC, an absorption band is obtained at around 410 nm which corresponds to the surface Plasmon resonance (SPR) absorption band of silver nanoparticles, indicating that the nanoparticles are present in the composite. Apart from the surface plasmon resonance (SPR) peak at around 410 nm, an additional wide-range absorption spread in the wide vis-region at wavelengths greater than 400 nm was observed in the rest of composite thin films. The wide-range absorption observed in the vis-region can be attributed to the characteristic localized surface plasmon resonance (LSPR) for the Ag NPs incorporated in the TiO_2 matrix (Ochoo *et al*., 2012; Xing *et al*., 2012).

UV-visible spectroscopy is a valuable tool for structural characterization of silver nanoparticles. It is well recognized that the absorbance of silver nanoparticles depends mainly upon size and shape (Elechiguerra *et al*., 2005). Zhou *et al*., (Zhao *et al*., 2012) reported that metal nanoparticles exhibit the absorption bands at 410nm because of surface plasmon resonance (SPR). In the case of Ag/TiO_2 composite thin film heat treated at 250ºC to 500ºC curves, it is observed that SPR shift to longer wavelength with peak position around 540 nm, which is decreasing with increasing heat treatment temperature, presumably due to the fact that the lack of agglomeration of Ag particles as illustrated in the FE-SEM images reported in our recent work. The enhancement in intensity of the absorption spectra is related to the increase in the number of Ag nanoparticles/crystallites in/on the film. The optical signature of this sample can be better understood in terms of the distribution of sizes and shapes observed in FE-SEM images reported recently (Daniel *et al*., 2015). The role of heat treating is therefore found to be responsible for the gradual enhancement in the surface states which changes the optical properties. Thus, the optical properties of the films depend strongly on the heat treatment temperature conditions.

Fig.1 Absorption spectra of Ag-NP/TiO_2 composite thin films: (a) low heat treatment temperature at 70, 250, 300, 400ºC, (b) high heat treatment temperature at 500, 600, 700, and 800ºC respectively

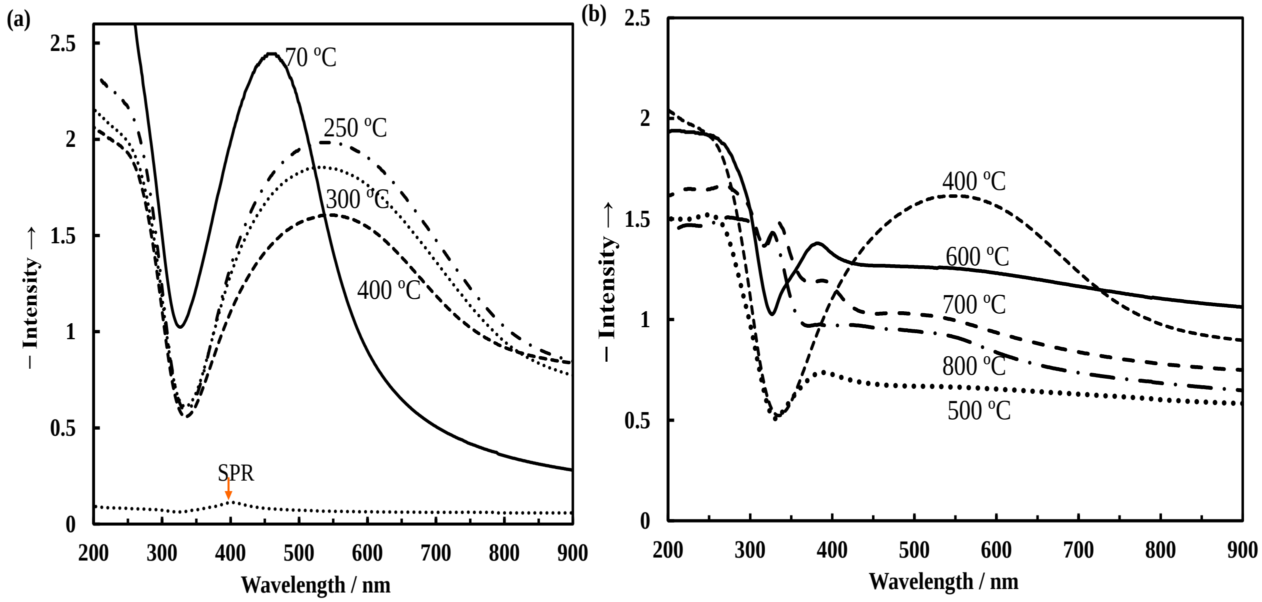

Fig.2 Electrical resistivity of Ag/TiO$_2$ composite thin film heat treated at different temperatures

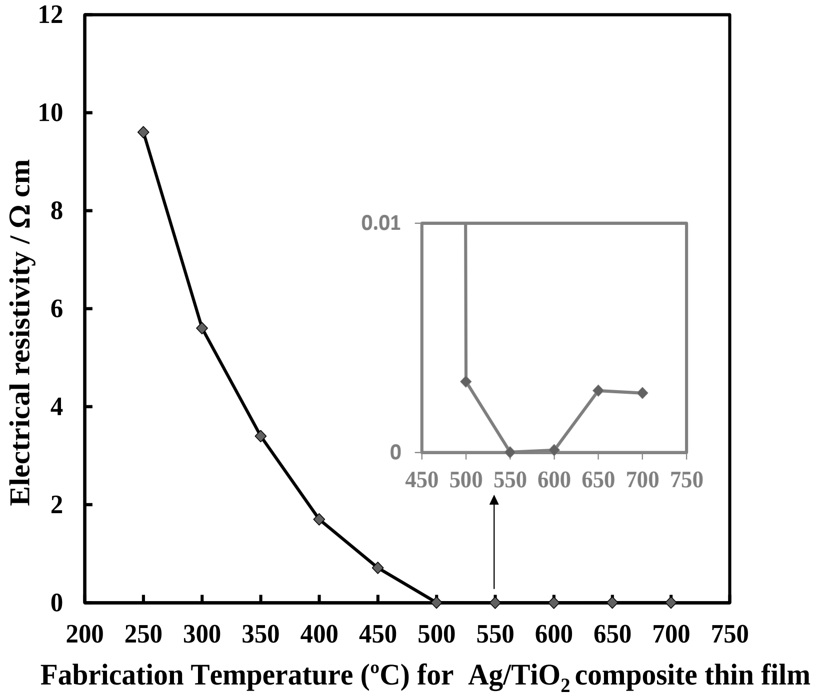

Fig.3 Photocurrent densities of Ag-NP/TiO$_2$ composite thin films: (a) low heat treatment temperature (b) high heat treatment temperature at 70, 250, 300, 400, 500, 600, 700, and 800ºC respectively

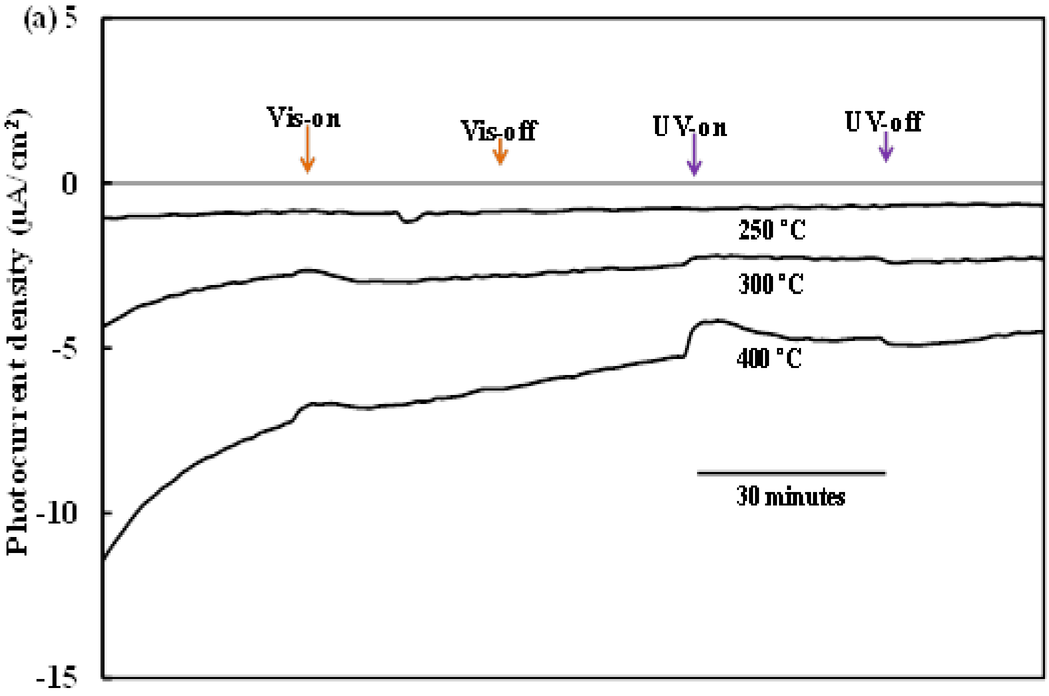

Table.1 Film thickness, averaged photocurrent density (APD), averaged dark current density (ADD) of the Ag/TiO$_2$ composite thin films fabricated at different het treatment temperatures (°C) on the quartz glass substrate

Notation	Film thickness	APD^{a,}		ADD^{a,}
		Vis-light	**UV-light**	**Dark**
	nm	**μA/cm^2**		
Ag/TiO$_2$250	-	-0.9 (2)	-0.7(3)	0.7(1)
Ag/TiO$_2$300	-	-2.1 (5)	-1.7(3)	2.1(4)
Ag/TiO$_2$400	270	-0.3 (2)	-5.2(7)	-5.9 (2)
Ag/TiO$_2$500	180	-7.1(6)	+0.1(3)	-0.9 (3)
Ag/TiO$_2$600	100	-9.2(3)	-2.7(4)	-1.0(3)
Ag/TiO$_2$700	90	-4.0(3)	-2.5(5)	-1.1(5)
Ag/TiO$_2$800	rough	-1.2 (2)	-0.1(1)	-0.2 (2)

The standard deviations are presented in parentheses.

Table.2 Young`s modulus and knoop hardness results obtained during the measurement of mechanical strength of the Ag-NP/TiO$_2$ composite thin films fabricated on a quartz glass

Notation	Young`s Modulus^a	KnoopHardness^a
°C	**Pa**	**Gpa**
Quartz glass	1.10×10^{11} (4)	8.41(3)
TiO$_2$600	7.47×10^{10} (2)	2.44 (4)
Ag/TiO$_2$500	6.70×10^{10} (4)	2.70 (1)
Ag/TiO$_2$600	8.49×10^{10} (6)	2.99 (1)
Ag/TiO$_2$700	9.87×10^{10} (2)	5.36 (5)

The standard deviations are presented in parentheses.

By considering the heat treatment temperature at low temperature region, that is 70–400ºC, the resistivity varies from 9.6×10^{00} to 1.7×10^{00} Ω cm. In this temperature region, some of the organic residues are still present in the thin films, hence are trapped inside the organic structure of the incomplete combustion. Thus, in this region, low conductivity is observed in the samples. As the temperature is further increased, from 400 to 600ºC, the resistance values of thin films are found to be decreased from 1.7×10^{00} to 1.1×10^{-4} Ω cm. This observation indicates that the Ag NP growth enhancing the grain–grain interaction in order to build the conductive network and crystallization as temperature increases. At the higher temperature region, 800ºC, the resistance values have decreased considerably, from 1.1×10^{-4} Ω cm to undetectable conductivity. The SEM image reported in our previous work shows that there exists crack on the thin films attributing to the effect of grain boundary. In general, the decrease in resistivity with heat treatment temperatures can be explained as follow: the Ag grains size increases with increase heat treatment temperature which lead to a decrease in Ag grain boundaries and hence resistivity. Larger silver grans size will provide higher surface contact between each other, improving electron migration. But, in thin film heat treated at 800°C case, it can be seen that the thin film is full of crack which affect the formation of the connecting network.

The enhanced near-field amplitude of localized surface plasmon resonance in the proximity of metal nanoparticles can boost the photo activity of the neighboring semiconductor, which has been proven and has attracted wide interest recently (Wang *et al*., 2012). One best way to prove this is simple by study the photo electrochemical properties of the doped semiconductor. The dark currents that were observed in all composite thin films heat treated at different temperature are an indicates that there is a redox potential between the counter Ag electrode and working electrode (composite thin films), hence chemical redox reactions occurred to the system. It has been reported that dark current can be generated in an electrochemical cell because of the recombination of charges by the reduction of species in the electrolyte or counter electrode, with species on the working electrode (Hu *et al*., 2003; Ishizawa *et al*., 1999). Daniel *et al.*, 2013 reported that cathodic dark current can be generated in composite thin films, given that the potential originating at the counter electrode is high enough to drive electrons flowing from the conductive composite thin film into the electrolyte.

The lower cathodic photocurrent for Ag/TiO_2 thin film under UV-light is due to the wide band gap of TiO_2 as a semiconductor that allowed it to absorb only UV light and to produce electron/hole pairs. However, rate of injection of these photo excited electrons into the conducting band of TiO_2 can be different because of different in electrical resistivity associated with Ag/TiO_2 fabricated at different temperature. Hence, it was difficult for the photo excited electrons to reach the TiO_2 surface, leading to an increase in extinction probability, which was unfavorable to the photo responsive activity of the thin films. Consequently, owing to the lower electrical resistivity associated with middle-Ag-level composite thin films (Fig. 2), coupled with the decrease in the intensity of SPR peaks, which can produce photo excited electrons in Ag NPs. The photo response of these composite thin films increased under vis-light irradiation as the photo excited electrons were injected easily into the conduction band of TiO_2. The large cathodic photocurrent density observed could be mainly due to LSPR (Warren *et al*., 2012).

When it come to the study of the mechanically strength of quartz glass, and Ag-NP/TiO_2 thin films whose heat treated at higher temperature (500, 600, and 700ºC), the values of Young`s modulus obtained by indentation methods are load dependent and highly sensitive to local defects (porosity, matrix grain pull-out, micro cracks, etc.) or structural in homogeneities (Špaková *et al*., 2008). Therefore, the decrease of the Young`s modulus values with an increasing of heat treatment temperature can be probably explained mostly by change in microstructure of the thin film. Never the rest, indentation of all the thin films heat treated at different temperature showed good mechanical results since their mechanical strength are between pure TiO_2 thin film and those of naked quartz glass substrate. Such good mechanical properties are necessary for their wide–spread application in different areas of industry such as solar cells.

In conclusion, the electrical conductivity, photo electrochemical and mechanical properties of Ag/TiO_2 composite thin films were investigated. The electrical resistivity of the films was of the order of 9.6×10^{00} to 10^{-4} Ω·cm with film thicknesses in the range 100–270 nm. The absorbance spectra of the synthesized Ag-NP/TiO_2 composite thin films decrease, and then increase in the visible region with heat treatment temperatures, indicating that the heat treatment temperatures are capable of sensitizing Ag/TiO_2 thin film, an understanding of the absorption spectra is of great importance to those studying Ag/TiO_2 thin films for photo electrochemical properties or other applications such as photovoltaics. For the study of photo electrochemical properties, photocurrent currents density were observed in all composite thin films heat treated at different temperature. This indicates that there is a redox potential between the counter Ag electrode and working electrode (composite thin films), hence chemical redox reactions occurred to the system. The mechanically strength of quartz glass, and Ag-NP/TiO_2 thin films whose heat treated at higher temperature (500, 600, and 700ºC) were examined in order to check their potential use in practical systems such as solar cells. The indentation of all these three composite thin films showed good mechanical results since their mechanical strength are between pure TiO_2 thin film and those of naked quartz glass substrate. Such good mechanical properties are necessary for their wide– spread application in different areas of industry.

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