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TRANSPARENT COATING OXIDE – INDIUM ZINC OXIDE AS CONDUCTIVE COATING: A REVIEW

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Abstract. This paper reviews the transparent conductive oxide with emphasis on indium doped zinc oxide (TCO-IZO). TCO has been growing faster year by year, especially in semiconductor, solar devices and electronic field. There are many methods that can be used to produce IZO coating such as dip coating, sol-gel, magnetron sputtering, thermal evaporation and atomic layer deposition. IZO has gained significant attention for transparent electrodes due to its good electrical conductivity, high visible transmittance in the range from 400 to 900 nm, large work function, excellent surface smoothness, and low deposition temperature. Many studies have been done on the IZO coating onto glass substrate, but there is still less study on the low processing temperature substrates such as polymers and natural fibres. Parameters such as coating thickness, annealing temperature and number of cycles have to be considered in order to achieve the desired electrical conductivity and optical properties of IZO.

1. INTRODUCTION

Transparent conducting oxide (TCO) and its mixtures have been extensively used in applications such as flat panel screen, in touch panels, solar cells, and other future devices which exhibit capability of transporting electrical charge and transmitting visible photon. They are also essentially used in electrochromic windows, oven windows and energy-efficient windows that release and trapped heat during winter and summer. TCO is a very useful material for transparent optoelectronics application due to its unique features of optical properties in the visible light region such as high optical transmittance over ~85% and wide optical band gap greater than 3 eV and controllable electrical conductivity such as carrier concentrations of at least 1020 cm^{-3} and resistivity of about 10^{-4} ohm.cm. It combines two important properties which are good electrical conduction and high transmission in the visible range of light (380-780 nm). Achieving high electrical conductivity and good optical transmission in TCO materials is crucial for most applications [1]. Most of the TCO materials are n-type semiconductors, but p-type TCO materials are researched and developed. Such TCO include: ZnO:Mg, ZnO:N, IZO, NiO, NiO:Li, $CuAlO_2$, Cu_2SrO_2, and $CuGaO_2$ thin films. At present, these materials have not yet found place in actual applications. For the purpose of improving TCO coating properties, new materials consisting of ternary compound oxides based on ZnO have been investigated. For example, ternary compound oxides such as In-doped

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ZnO (IZO), Al-doped ZnO (AZO), Ti-doped ZnO (TZO), and Si-doped ZnO (SZO) have attracted considerable attention as alternative materials for ITO. Indium-tin oxide (ITO) films are the most extensively studied and commonly used as TCO film since its film has high electrical conductivity and high transparency as well as ease of fabrication on glass substrates at a substrate temperature over 473K. However, amorphous ITO films exhibit a decrease in electrical conductivity and optical transparency with time and have a very poor chemical stability. These problems can be solved by using IZO films that can be deposited using variety of techniques and exhibit excellent chemical stability with highly electrical conductivity and optical transparency [2-5].

Recently, zinc oxide or impurity (B, Al, Ga, In, and Zr) doped zinc oxide films have been investigated because of its nontoxicity, inexpensiveness and availability. The development of IZO coating onto low processing temperature material such as natural fiber is challenging yet promising as it has not been covered by previous researcher. The recent increased demand and emerging interest in the development of electrically conductive polymer composites make the prospect of IZO coating on natural fiber to be very interesting as it has the potential to be utilized as conductive filler and reinforcement.

2. TCO MATERIALS – ZINC OXIDE (ZnO)

ZnO has direct band gap semiconductor of 3.27 eV and has been recognized for its promising applications in blue/UV optoelectronics, spintronic devices, surface acoustic wave devices and sensor applications. El Yamny et al. (2012) reported that undoped of ZnO thin films are not stable at high temperatures and can be improved by introducing impurities into the ZnO host lattice, which dramatically reduces this disadvantage [6]. Doping of ZnO with elements of Group XIII such as B, Al, In, Ga increases the conductivity of the ZnO thin films. The doping process is performed by means of replacing Zn^{2+} atoms with impurity of higher valence. It is worth mentioning that the efficiency of the dopant element depends on its electronegativity and difference in the ionic radius as compared to zinc. Aluminum and indium are well-known dopants for n-type ZnO and have been studied to some extent [7]. Transparent conducting electrodes for thin solar cells has identified zinc oxide as one of the best option due to its high chemical stability against reducing environment [8], textured surface and the simultaneous concurrence of high transparency in the visible region as well as highly conductive. Some other advantages are wide material availability, non-toxicity and easy handling that makes ZnO suitable for wide area of applications [9]. In comparison with indium tin oxide (ITO), ITO has been mainly used as TCO anode because of its high conductivity, large work function and transparency over the visible range [10]. Pure ZnO has high resistivity and well-known as n-type semiconductor. Addition of indium impurity will substitute Zn because of the similarity in ionic radius. Since Indium has one more electron than Zn, it acts as donor impurity and creates an n-semiconductor [1]. Doping directly affects carrier concentration and influences conductivity in turn. Although introducing impurities is necessary, but the concentration has a limitation; otherwise, a very high doping level will result in high free carrier absorption, high plasma resonance reflectivity, and low visible wavelength transparency. IZO has significant attention for transparent electrodes due to its good electrical conductivity, high visible transmittance in the range from 400 to 900 nm, large work function, excellent surface smoothness, and low deposition temperature [11].

Indium doped zinc oxide film was deposited from mixed precursor of indium and zinc salts in the atomic percent ratio, In/Zn = 1% – 9% using dip coating method. As a result, doping range between 1% - 5% demonstrated the lowest electrical resistivity, indicating an increase in donor concentration, which is attractive for many optical and electrical applications. The donor action of indium compensated the grain boundary scattering leading to decrease in film resistivity. As the doping concentration increased, the grain size decreased due to deformation. Though the carrier concentration is expected to increase by indium doping, the smaller grain size enhances the grain boundary scattering and hence leading to an increase in the resistance at higher indium content [6]. Increased dopant to more than 6 at.% changed the n-type semiconductor to p-type [1] as previously studied using 2, 4, 6, 8, 16, and 32 at.% of dopant concentration. Considerable point in [1] is that of converting n-type to p-type semiconductor with increasing impurity (higher than 6%). It was found that conductivity initially decreased as converting semiconductor (n-type to p-type) and decreasing its carrier concentration. But following the increasing impurity, the carrier concentration increases so that the lowest resistivity was observed with impurity of 32% [1].

3. TYPE OF SUBSTRATES

Recently, transparent conductive films have been prepared on plenty of different substrates, such as glass, sapphire, and polymer as studied by previ-

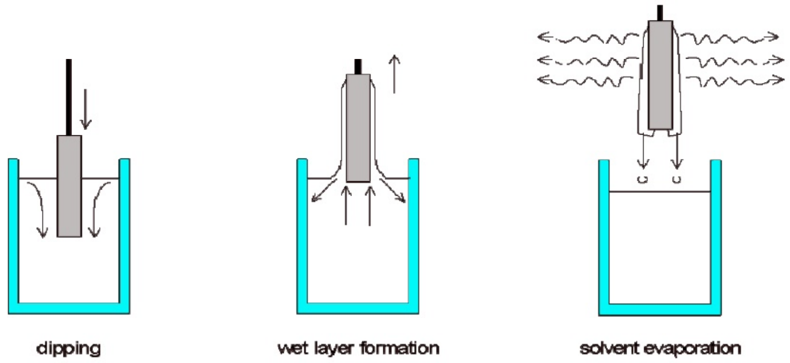


Fig.1. Stages of the dip coating process.

ous researchers [1,12-14]. Each substrate has their own specific properties that may cater for different needs. For example, glass substrates are heavy and brittle, whereas polymeric substrates such polyethylene terephthalate (PET) are lightweight, unbreakable and conveniently portable. Substrates nature alone may affect the microstructures, crystallization, morphology and electrical properties of films. The interest in natural fiber is rapidly growing either in terms of fundamental research or actual industrial application. They are renewable, cheap, completely or partially recyclable, and biodegradable. Plant-derived natural fibres consist of cellulose, hemicelluloses, lignin, pectin and waxy substances. Cellulose gives strength, stiffness and structural stability of the fibre, and its own cell geometry is responsible for the determination of mechanical properties of fibres. Their availability, renewability, low density, and price as well as satisfactory mechanical properties makes them an attractive ecological alternative to glass, carbon and man-made fibers used in the manufacturing of composites [15]. However, there are several challenges presented by natural fibers such as large variability of mechanical properties [16,17], lower ultimate strength, lower elongation, problems with nozzle flow in injection molding machines and bubbles in the product [18]. Additionally, most natural fibers have low degradation temperatures (~200 °C), which make them incompatible with the high curing temperatures or any other processes that involve high temperature. This also restricts natural fiber composites to relatively low temperature applications [19].

4. TCO DEPOSITION TECHNIQUE

There are various techniques used for TCO deposition process such as dip coating, sputtering, chemical vapour deposition and electron beam evaporation (spray pyrolysis). It also can be produced by RF magnetron sputtering, sol-gel process, electrochemical deposition, molecular beam epitaxy (MBE), pulsed laser deposition (PLD) and metal organic chemical vapor deposition [6]. Due to the very high deposition rate, electron beam evaporation method has potential industrial application [20]. Sputtering can be used for coating large area of substrates at high rate and with competitive costs compared to other methods [21]. TCO based on indium doped zinc oxide films in the nano scales have been successfully prepared using combination between dip coating and thermal decomposition process [6]. Thus, the deposition on organic substrates such as polyimide (PI) and even on flexible organic substrates such as polypropylene adipate (PPA) is possible [22]. Reactive radio frequency sputtering (rf MS) is commonly utilized for the sputter deposition of TCO materials [23].

In other research, Rezaee et al. (2009) has applied sol gel method to produce indium doped zinc oxide onto glass substrate, because of high purity, homogenous and large area film at low temperatures [1]. Thamarai et al. (2014) claimed that dip coating method is extremely attractive due to its advantageous features over other thin film deposition technique, such as its simple low temperature, low cost, easy coating of large surfaces and low evaporation temperature [24]. Dip coating deposition process are known as an economical deposition process, faster and ease of parameter control process as shown in Fig. 1. Basically, the process may be separated into three important technical stages: (1) Immersion and dwell time, (2) Deposition and drainage, (3) Evaporation [25]. Meanwhile, Fig. 2 shows the details of the flow patterns (streamlines) during the dip-coating process where U_0 is the withdrawal speed, S is the stagnation point, δ the boundary layer and h_0 is the thickness of the entrained fluid film on the substrate [26]. El Yamny

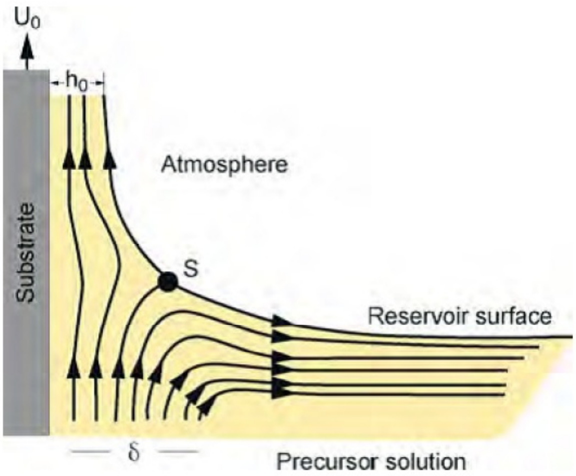


Fig. 2. Flow pattern during dip coating process, adapted from [25].

et al. (2012) has successfully studied the Zn:In by dip coating with the aim to produce low cost, wide area productivity and good film quality, in order to compete with other sophisticated preparation techniques. It was reported that dipping rate in the range of 2 - 38 mm/s as the most suitable for good film quality while dipping rate in the range of 30 - 38 mm/s produced thicker films in lower deposition cycles. The higher dipping rate produces films with lower transparency while the small deposition rate requires large number of deposition cycles [6].

5. COATING PARAMETERS

5.1. Thickness

Thickness of film coating, which ranges from ~50 to ~2000 nm, is among the factors that determine the electrical and optical properties of ZnO-based TCO films. The electrical conductivity becomes better while transmittance may become poorer as the film thickness increases [27]. Kuo et al. (2011) reported that when the thickness is over the critical value, the excess internal stress causes structural discontinuity by the bifurcated orientation, and it might also crack the film; these both restrict the carrier mobility and reduce the conductivity [28]. Coating thickness is crucial as it is directly related to the resistivity of the ZnO thin film. The crystallite's size and orientation which depend on the film thickness play a big role in controlling the electrical conductivity of the polycrystalline ZnO film [29]. It is found that the crystalline quality and the grain size increases with film thickness. Resistivity first decreases for thicknesses up to 41.2 nm, due to the increase in carrier mobility. For films above 50 nm, the resistivity increases due to the increase of trapped carriers in the grain boundary.

The resistivity as well as films thickness are affected by dipping rate. Fig. 3 represents the dependence of the film thickness on dipping rate. As dipping rate increases, the amount of precursor deposited on the substrate increases then evaporation of the solvent occurs before dropping back to the precursor. In addition, at lower dipping rate, better film quality and clarity are produced. Furthermore, the increase of dipping rate strongly decreases the resistivity of the film thickness. This phenomenon is due to compactness of the grains by considerable higher deposited amount of the precursor. At a very high dipping rate, milky and powdered films are obtained, while at a very low dipping rate, both the film thickness and compactness of the grains become too low to conduct an electrical current. The dipping rate in the range of 2 - 40 mm/s is considered as the optimal range for better, clear and electrically conducting films [6].

5.2. Growth temperature

To realize transparency and conductivity in low processing material such as polymer and natural fibre, it is therefore necessary to develop TCO that can be prepared at low temperature (<200 °C) while maintaining its good optical properties. Low growth temperature is required for coating on various flexible substrates such as OLED, PMMA, and natural fibre. Unfortunately, at low growth temperature, the resulted films always show poor properties. However, this association does not mean that all hope for low-temperature growth is lost. In recent years, successful efforts have been reported regarding the

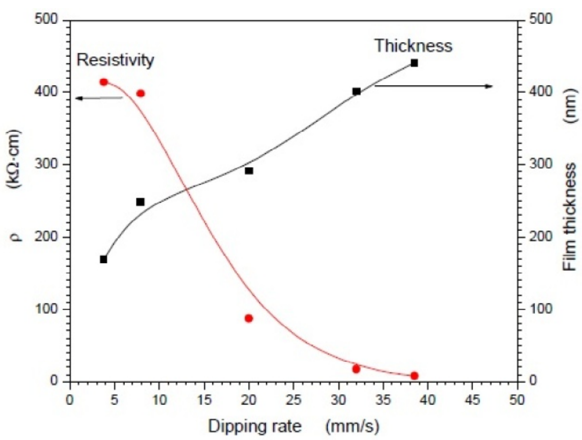


Fig. 3. Dipping rate dependence of electrical resistivity and film thickness, adapted from [6].

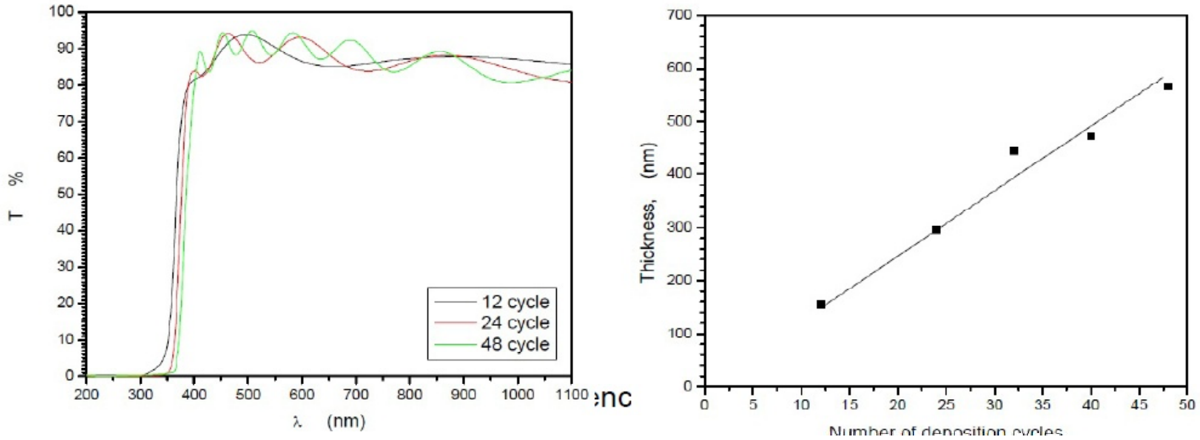


Fig. 4. a) Deposition cycles dependence of the optical transmittance of ZnO:In; b) Deposition cycles dependence of the film thickness, adapted from [6].

growth of high quality films at low temperature on flexible substrates and they attracted extensive attention owing to its rather low processing cost and many unique applications [30,31]. For example, Clatot et al. (2011) have successfully deposited Si-doped zinc oxide (SZO, Si 3 wt.%) thin films at a relatively low temperature (≤150 °C) by pulsed laser deposition (PLD) [32]. Recrystallization and growth of films reduced internal imperfections, resulting in higher conductivity, Hall mobility (increased from 6.56 to 10.12 cm^2/Vs), and higher transmittance [33,34]. It is also worthy to note that the merit of no damaging on the underlying substrates makes this technology promising in various low temperature applications [35].

Indium zinc oxide (IZO) is one of the best materials that exhibits good transparency in the visible range, low resistivity, and high mobility [36-39]. Annealing temperature has a direct effect on the electrical properties and surface morphology of IZO coating. Based on Edynoor et al. (2017), they were claimed that kenaf fibre was successfully coated with indium zinc oxide (IZO) by dip coating method with an emphasis on its morphology and electrical conductivity. Moreover, it was achieved by low annealing process of 150 °C for 4 h soaking time [36]. Upon annealing at a temperature range of 500 to 1000 °C, the thin film crystallizes into polycrystalline In_2O_3(ZnO). The lowest electrical resistivity was obtained when the annealing temperature was 700 °C at 2 Ω cm. As the temperature increases, the amount of oxygen desorption increases, and free carriers in the thin films also increase [14]. It was reported that with the increase of the annealing temperature of IZO (In_2O_3:ZnO = 90:10 wt.%) from 100 to 300 °C, the carrier concentration decreased and the resistivity increased. The average transmittance of IZO thin films decreased slightly with increasing annealing temperature, and it had amorphous structure [40]. During annealing treatment, the absorbed oxygen reduced the oxygen vacancies in the film, and lead to decreased carrier density and increased mobility. When annealed at temperatures of 200, 300, 400 °C for 1 h in open air, the structure of ITO changed from amorphous to polycrystalline, but that of IZO continued to be amorphous. The transmittance of both films was improved while the resistivity decreased with the increase of annealing temperature at low oxygen content [11]. Upon annealing at a temperature range of 500 to 1000 °C, IZO thin film which was prepared by using zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$) and indium acetate ($In(CH_3COO)_3$) as starting precursors, and 2-methoxyethanol with 1-propanol as solvents crystallized into In_2O_3:ZnO polycrystalline [14].

5.3. Number of cycles

Number of cycles is another parameter that should be taken into consideration since it correlates with the thickness of the TCO film. As claimed by El Yamny et al. (2012) increasing the film thickness by increasing the deposition cycles has a limitation that the film quality and transparency is decreased. Figs. 4a and 4b show the deposition cycles dependence of the optical transmittance and film thickness of In_2O_3:ZnO [2]. Thamarai et al. (2014) also revealed that optical transmittance was found to decrease from 65% to 55% and 52% with the increase of growth time from 3 to 4 and 5 hours, re-

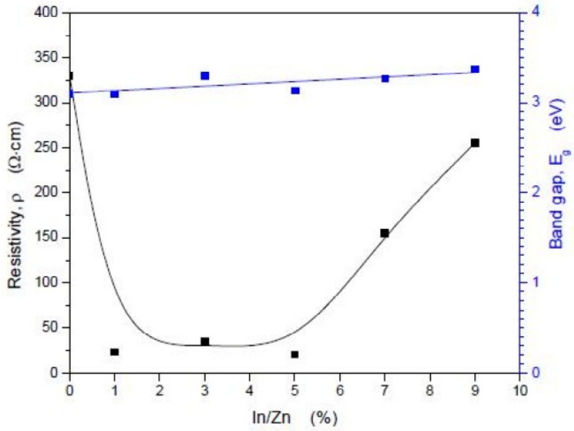


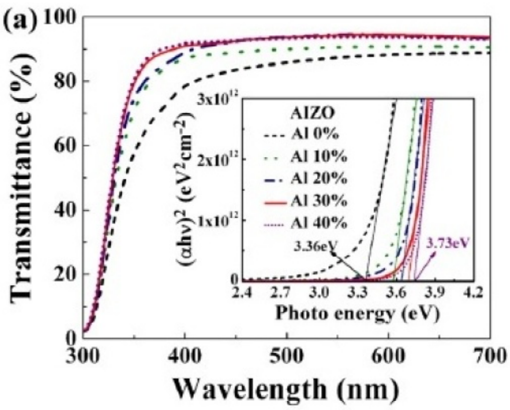
Fig. 5. In/Zn percentage (0-9%) dependence of film resistivity and band gap energy, adapted from [6].

spectively. It also clearly indicated that as growth time increases the band gap decreases. The decrease in band gap of ZnO films may be attributed to the improvement in the crystalline quality of the films along with the reduction in porosity and increase in grain size [24].

6. PROPERTIES OF IZO

6.1. Electrical properties

The electrical conductivity is due to doping either by oxygen vacancies or by extrinsic dopants. Rezaee et al. (2009) found that by increasing impurity higher than 6%, it leads to the change of the n-type semiconductor to p-type. This is probably due to the decrease in donor property of indium [1]. Electrical conductivity becomes better as the film thickness increases as more photons are adsorbed when the thickness increase [27]. Doping of ZnO by indium in the range 1 - 9% shifts the absorption edge towards the shorter wavelength. The shift in the absorption edge was suggested to be attributed to carrier concentrations, carrier distributions and defects presented in the film [6]. The carrier concentration increased sharply and reached maximum value at 700 °C, and then started to decrease. Free carriers of IZO originate from donor sites associated with oxygen vacancies so that the curve of the carrier concentration could be explained by oxygen vacancies. As the temperature increases, the amount of oxygen desorption increased, and free carriers in the thin films also increased. However, the situation was reversed over 700 °C because of oxygen diffusion into the thin films. As the temperature increase, the diffusion rate increased and the oxygen compensation also increased [14]. The optimal values of indium were found to be around 1.5 - 5%, at which donor concentration increased and the ZnO showed the lowest resistivity as demonstrated in Fig. 5. The donor action of indium compensates the grain boundary scattering and contributes to the decrease in film resistivity. As the doping concentration increases, the grain size decreases due to deformation. Although the carrier concentration is expected to increase with indium doping, the smaller grain size enhances the grain boundary scattering, thus increases the resistance at higher indium content. Furthermore, Barquinha et al. (2007) studied IZO annealing in three different atmospheres, i.e., vacuum, air and oxygen. It was described that for the conductive thin films, only the oxygen atmosphere was critical, leading to an increase of the electrical resistivity of more than one

a) 

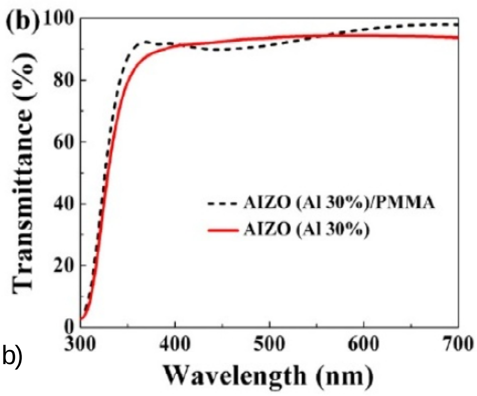
b) 

Fig. 6. (a) Optical transmittance spectra of α-AIZO thin films dip-coated with different Al content. The inset shows plot of $(\alpha h\nu)^2$ versus $h\nu$. (b) Typical optical transmittance spectra of α-AIZO (Al 30%) + PMMA double layers, adapted from [43].

order of magnitude, for temperatures of 250 °C and above [41].

6.2. Optical properties

It is proved by previous researchers that the optical transmittance spectra showed a very good transmittance of 99.5% within the wavelength of 540 nm for the film doped with 2 wt.% In onto glass substrate. Increased dopant concentration would reduce transmittance of visual spectra of samples. This is due to incident diffraction caused by the indium atoms surrounded by ZnO indicating that indium atom is slightly larger than Zn and crystal imperfection [1]. Moss-Burstein effect explained that the optical band-gap is shifted towards short wavelengths with high carrier concentration values. This is contrast to Lee et al. (2012) which mentioned that the absorption edge shifted towards long wavelengths with increased concentration [14]. This might be due to the phenomenon of 'quantum confinement effect', which is caused by the grain size of films [42]. In fact, the grain size of the thin films was comparatively small in that range of temperature. In other research, Yue et al. (2013) observed that the transparency of the IZO film was obviously improved by incorporation of Al. AIZO are highly transparent in the visible range (400-700nm) with average > 90% except of Al-free IZO thin film. Fig. 6 shows the optical transmittance spectra of the AIZO films as a function of the Al content [43].

7. CONCLUSIONS

TCO-IZO development as reported in various related papers has been reviewed. Good TCO should has wide optical band gap (>3.5 eV), low electrical resistivity (<10^{-3} W.cm) and high optical transmittance (>80%) in the visible region. Doping concentration plays an important role because it affects carrier concentration and influences conductivity in turn. Preparation methods, substrate type, film thickness, growth (annealing) temperature, number of cycles and ambient atmosphere are factors that determine the electrical, optical and structural properties of TCO. Among these, film thickness is crucial and need to be controlled as it is directly related to resistivity. Furthermore, growth temperature needs to be considered for the substrate having low processing temperature such as polymers and natural fibres. As IZO coating onto glasses and polymer substrates using various type of deposition technique have been widely studied, the author has put an effort to study coating onto natural fibre substrate as it has not been covered yet. The coating properties are unknown and its future potential requires more investigation. As the interest on conductive polymer composites is increasing, the newly natural fibre based TCO-IZO coating can be utilized as conductive filler or reinforcement in polymer composites.

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