Comparison of RF and Pulsed Magnetron Sputtering for the Deposition of AZO Thin Films on PET

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AZO thin films (around 200 nm thick) were grown on polyethylene terephthalate (PET) at room temperature. The plasma was activated using a 13.56 MHz (RF) or a 15 kHz pulsed (PMS) source at a power of 60 W. Optical reflection and transmittance were measured using a UV-Vis-NIR spectrometer over the wavelengths from 190 nm to 2500 nm. All samples show average transmittances greater than 83% in the visible region. The electrical resistivity was measured by the linear four-point probe method to be around 0.001 Ω cm for 200 nm-thick AZO films grown by PMS. XRD results indicated that the films had a hexagonal wurtzite structure and were preferentially oriented in the (002) plane. The surface morphology of the AZO thin films was characterized using Scanning Electron Microscopy (SEM); film chemical composition was studied using Energy Dispersive X-ray Spectroscopy (EDS). For this, an EDS coupled to the Scanning Electron Microscope was used. Only for films grown by PMS were no cracks observed.

Keywords: TCO, zinc oxide, PET, magnetron sputtering.

1. Introduction

Films of Transparent Conducting Oxide (TCO) have been used, amongst others, as transparent electrodes, in photovoltaic cells, light-emitting films, and intelligent windows^{1.4}. Nowadays, the most used TCO is Indium Tin Oxide (ITO). This material, however, is expensive to produce owing to the relatively low abundance of In in the Earth's crust. Moreover, In is a toxic element, which restricts its use in the usual plasma processes used to produce electronic devices.

Consequently, alternative TCOs are being sought that could substitute ITO, especially in technologies that require large quantities of material, for example, in photovoltaic cells. Thus, zinc oxide doped with Al (AZO) is a promising TCO because of its distinct characteristics, such as being a naturally abundant, non-toxic, element, with a low production cost, and being compatible with chemical and physical processing for the production of opto-electronic devices. Regarding its electrical properties, new forms of synthesis have been studied to obtain AZO films as conductive as ITO, while maintaining its optical transparency above 80% in the visible region.

Recently, the use of polymeric substrates for the fabrication of light-emitting devices⁵ and even of photovoltaic cells⁶ has been consolidating scientifically and industrially. Diverse flexible polymers, such as polyamide, have been studied as substrates for the fabrication of opto-electronic devices. Polyethylene Terephthalate (PET) has received considerable attention for several reasons. First, compared to polyamide (PI), PET has greater optical transparency and mechanical resistance, and lower fabrication cost. Second, PET recycled from food packaging may be used, further reducing the production cost and contributing to environmental preservation.

Diverse methods such as sol-gel, thermal vaporization, PLD and magnetron sputtering have been employed to synthesize TCOs. In these deposition methods^{7,8}, desired resistivities were observed for TCOs, but required the use of temperatures greater than RT (room temperature). Owing to the degradation of the polymeric matrix above the melting point (for example, 80° C for PET and 250° C for polyamide), or by collisions with energetic species, however, there are some limitations on the choice of synthesis parameters of TCO. Thus, the synthesis of TCO films on polymeric substrates requires process parameters that do not degrade the substrate, allow good adhesion, generate little film/substrate stress, and yet maintain the desired optical and electrical properties⁹⁻¹³.

In this context, *magnetron sputtering*, in addition to excellent reproducibility and low production cost in large scale, also allows the synthesis of thin films while maintaining the substrate at room temperature¹⁴⁻¹⁶. Even in this case, however, the deposition parameters must be finely adjusted to obtain a transparent, electrically conducting film, well-adhered to the substrate. As observed in several studies¹⁷⁻¹⁹, devices containing TCO films combined with flexible substrates can be obtained by exploiting the advantages of sputtering already mentioned.

According to Shen et al.¹⁰ and Koidis et al.²⁰, AZO/PET films present an electrical resistivity of $10^{-3} \Omega$ cm when using higher plasma powers. However, there was a reduction in optical transparency caused mainly by the breaking of chemical bonds of the PET produced by the collision of energetic particles produced in the plasma. Other important aspects are the production of cracks and low adhesion to the

substrate. Cracks may be generated by factors such as high deposition rates or collisions with energetic particles during the deposition as well as mechanical stress or morphological structures of the deposited film. In turn, according to Fortunato et al.²¹, cracks cause a reduction in the electrical conductivity of the TCO film.

For these reasons, the power applied to the plasma and the type of power supply are crucial factors for the successful deposition of AZO on polymeric substrates or organic devices by *magnetron sputtering*. Indeed, Lin et al.²² reported that the electrical resistivity of ITO/PES films remained at $1x10^{-3}$ Ωcm even at low deposition powers when using a pulsed supply. The same behavior does not occur with films produced using a RF supply.

Hence this work compares the synthesis of AZO films on PET by *magnetron sputtering* at room temperature, using a RF supply (13.56 MHz) and a pulsed supply of frequency 15 kHz (PMS). In addition, the formation of cracks and the electrical stability with ageing were assessed for AZO films of different thicknesses deposited on PET.

Several TCO's require high deposition temperatures, which makes their application onto polymeric substrates unfeasible²³. The results of the present study could help solve this problem. Deposition on polymeric substrates could expand the possibilities of new technologies such as Flexible Electronic Devices (FEDs), organic photovoltaics, organic light-emitting diodes, etc²⁰. Also, it could facilitate the production and optimize the cost on an industrial scale by using roll-to-roll manufacturing methods²⁴.

2. Experimental

Rectangular PET substrates, 35 mm x 25 mm, and 0.1 mm thickness were used. For 10 min. for each stage, substrates were cleaned in an ultrasonic bath of distilled water, and then washed in isopropanol. A conductive AZO (ZnO with 2 wt.% of Al₂O₃ 99.9%) target of 3 inches diameter was used. Film deposition was undertaken using RF (13.56 MHz) and a pulsed supply (15 kHz). The target-sample distance ($d_{T,S}$) was 4 and 5 cm. In both cases, the argon pressure was fixed at 3 mTorr and the power maintained at 60 W for 30 min.

The thickness of the AZO/PET films was measured using a DEKTAK 150 profilometer. Electrical properties were determined using the linear four-point method. The optical transmittance and reflectance measurements were made using a Perkin Elmer Uv-Vis-NIR Lambda 750 Spectrometer over the wavelength range of 190 to 2700 nm. The chemical composition of the films was estimated using EDS with an accessory attached to a JSM-6010LA Scanning Electron Microscope. The density of cracks at the microscale in the films was also assessed using EDS.

Structural properties were analyzed using X-ray diffraction with a Panalytical X'Pert Powder diffractometer using Cu K_a radiation at a step-rate of 0.02 (°), an integration time of 6 s, and scan from 20 to 80° in grazing angle incidence (2°). Crystallite grain size (D) was estimated using the Scherrer equation²⁵

$$D = 0.9\lambda / \beta.\cos(\theta) \tag{1}$$

where λ = 0.154056 nm, β and θ are experimental values of the FWHM and (002) peak position of spectra of AZO films. The residual tension (stress) in the AZO/PET films was also assessed using the following equation²⁶:

$$\varepsilon = \left(C_{film} - C_{bulk}\right) / C_{bulk} \tag{2}$$

where C_{film} is the lattice parameter obtained from the diffractogram of the synthesized film and C_{bulk} is the lattice parameter of bulk ZnO.

3. Results and Discussion

Table 1 presents the thickness, mean optical transmittance in the 400 to 700 nm wavelength range and electrical resistivity of the AZO/PET films as a function of the type of supply (RF and PMS) and different target-substrate distances. The optical transmittance of the AZO/PET was greater than 80%, independent of the deposition parameters. Film electrical resistivity was around $10^{-2} \Omega cm$ with the RF supply and $10^{-3} \Omega cm$ with PMS. The decrease in resistivity obtained using a PMS power supply is caused by the absence of cracks (that is revealed by SEM) and to the high concentration of carbon (as revealed by EDS) compared to that of films deposited using RF. In addition, the electrical resistivity increased as the target-substrate distance increased from 4 cm to 5 cm.

Figure 1 shows the transmittance and reflectance spectra of the PET substrate and the AZO/PET films described in Table 1. Above 1250 nm, the transmittance spectra exhibit a

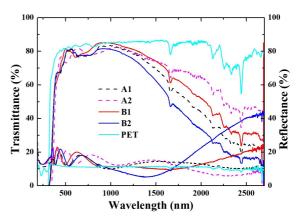


Figure 1. Optical transmittance and reflectance spectra of the PET substrate and the AZO/PET samples described in Table 1.

Table 1. Type of supply, target-substrate distance, thickness, optical transmittance and electrical resistivity of the samples studied.

Sample	Supply	d _{T-S} (cm)	t (nm)	T (%)	ρ (m Ω .cm)
A1	RF	5	260	80	38
A2	RF	4	272	80	17
B1	PMS	5	193	81	7.0
B2	PMS	4	250	83	2.5

reduction typical of TCOs. Below 370 nm there is absorption owing to interband transitions in AZO. In the visible region, the transmittance remains ~80%, while the optical reflectance is around 15%. Interference fringes are present. Compared with the PET spectra, note that AZO is almost transparent in this region of the spectrum. These films have greater transmittances than similar films on polymeric substrates reported in the literature^{4,10,27}.

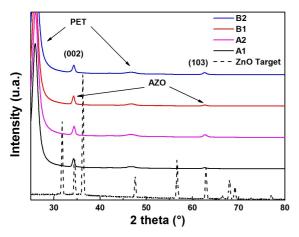


Figure 2. X-ray diffractogram of bulk ZnO bulk and of the AZO films deposited on PET. The arrows indicate the DRX peaks of the PET substrate and the (002) and (103) planes of the AZO films.

Figure 2 shows X-ray diffractogram of the AZO/PET films described in Table 1. For reference, the diffractogram of bulk ZnO is also shown. The peaks at 25.88° and 48° are related to the PET substrate, as indicated in earlier work^{4,12,24}. The peaks near 34° and 62° refer to the (002) and (103) of the AZO film, respectively. Note that all the films are polycrystalline, having a hexagonal wurtzite structure typical of ZnO films^{28,29}. Moreover, the presence of the prominent peak related to the (002) plane, indicates that the films have a preferential orientation in this direction, as also observed in the literature^{12,23,30}.

Table 2 shows the angular values (20) and the FWHM (β) of the (002) peaks of the spectra of the AZO films, as well as the estimated values of the crystallite size (D) and the residual tension (ϵ) of the film/substrate. The data indicate that the PMS process reduces slightly the crystallite size. For all the samples, the residual tension is considerably reduced from -1.01 to -0.48 for sample A1 and B2. The negative values indicate compressive stress. This reduction may be related to the absence of cracks in the film deposited by PMS, as revealed by MEV/EDS analyses to be discussed below.

Figure 3 shows scanning electron micrographs, taken in secondary electron mode, of the surfaces of the AZO/PET films. The formation of cracks is visible in the films produced by RF, especially for the target-substrate distance of 4 cm. For the films produced by PMS, no cracks are visible, at least on the same scale. Figure 4a shows a micrograph of sample A2 marked with the three positions where EDS

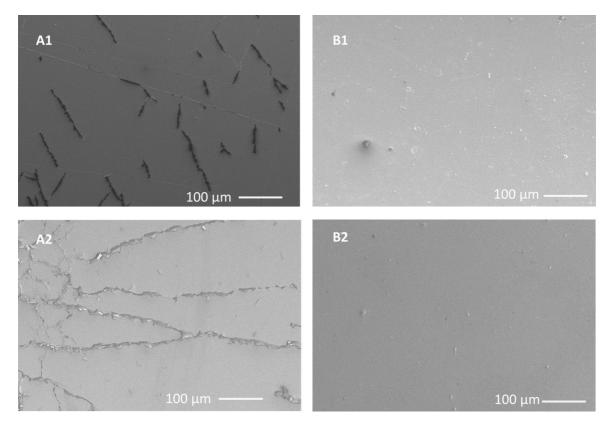


Figure 3. Micrographs (obtained by SEM) of AZO/PET films deposited by RF and PMS, with target-substrate distances of 5 and 4 cm. For the films deposited using RF, the presence of cracks is evident, which affects the adhesion to the substrate and the electrical conductivity of the AZO film.

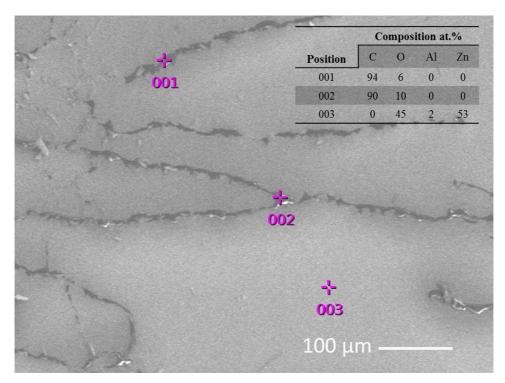


Figure 4. Scanning electron micrograph of the AZO/PET films deposited by RF (sample A2). Three positions are indicated where EDS measurements were made. The 001 and 002 positions, which are above cracks, exhibit high concentrations of C, owing to the PET substrate. In contrast, at position 003 only Zn, O and Al, which originate form AZO, are detected.

Table 2. Position angles (2 θ) and FWHM (β) of the (002) peaks of the spectra of the AZO films and estimated values of the crystallite size (D) and the residual tension (ϵ) of the film/substrate. The 2 θ angle of the (002) peak and the FWHM of bulk ZnO bulk were 34.48° and 0.32°, respectively.

Sample	2θ (°)	FWHM (°)	D (nm)	3
A1	34.26±1.2x10-3	0.55±4.4x10-3	15.1±9.8x10-5	- 1.01±3.3x10 ⁻²
A2	34.26±1.3x10-3	0.55±4.5x10-3	15.2±9.9x10-5	- 1.01±3.2x10 ⁻²
B1	34.28±4.5x10-3	0.61±15x10-3	13.6±3.2x10-4	- 0.81±6.5x10 ⁻²
B2	34.35±2.5x10-3	0.59±8.5x10-3	14.1±1.8x10-4	- 0.48±1.6x10 ⁻²

measurements were made. The 001 and 002 positions are above cracks, while position 003 is above a homogeneous region of the film.

Figure 4b shows that the 001 and 002 positions exhibit a high carbon concentration, which derives from the PET substrate. This shows that in the cracks, the film is completely separate, revealing the polymeric substrate and causing an increase in the electrical resistance of the AZO film.

At the 003 position, only Zn, Al and O are revealed, which are characteristic of the AZO. Furthermore, the composition of the AZO film is compatible with the EDS values (at.%) for the ceramic target: Zn=55.0, Al=1.7 and O=43.3. There are slight increases in the concentrations of O and Al, which are consistent with their greater bond energies³¹⁻³³.

Such results also indicate that EDS measurements may be used to quantify the presence of cracks, via de C concentration. For this, EDS area analyses were carried out with an electron beam over a typical area of 1 mm x 1 mm. The data obtained are shown in Table 3. From Figure 4, the C concentration represents an estimate of the crack density (more precisely the area of the exposed substrate) along the AZO film.

The films synthesized by PMS present a much lower density of cracks than films produced using RF. Indeed, the EDS data indicate that sample B2 is free of cracks. Moreover, this film has an optical transmittance of 83%, an electrical resistivity of 2.5 x $10^{-3} \Omega$ cm, and the lowest estimated residual tension (-0.48). Thus it may be concluded that the PMS synthesis at a target-substrate distance of 4 cm produced superior AZO/PET films both in electrical properties and in film/substrate adhesion.

Our result shows an advance in comparison with those presented by Tsay and Pai³⁴ who found a resistivity of $3.7 \times 10^{-2} \Omega$ cm using a deposition temperature of 135° C.

To investigate the stability of the films upon ageing, films were deposited using PMS under the same conditions as those used for sample B2 (P=60W, $d_{r.s}$ =4cm at room temperature) for deposition times of 5 min (B3) and 30 min (B4). The film thicknesses, measured using profilometry, were 51±3 nm and 200±10 nm, respectively. Figure 5 represents the optical transmittance and reflectance spectra

Table 3. EDS area measurements over an area of 1 mm x 1mm of the AZO/PET films. From Figure 4, the C concentration represents an estimate of the density of cracks along the AZO film.

Composition (at.%) ±3%							
Sample	С	0	Al	Zn			
A1	10.9	42.6	1.4	46.2			
A2	22.8	38.9	1.2	43.8			
B1	2.2	44	1.5	52.3			
B2	0	44.9	1.6	52.4			

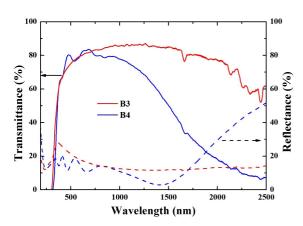


Figure 5. Transmittance and reflectance spectra of AZO/PET films deposited under the conditions of sample B2 (PMS and d_{T-S} =4cm) for 5 min (B3) and 30 min (B4).

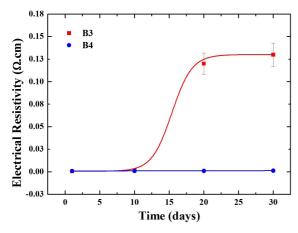


Figure 6. Electrical resistivity of the AZO films obtained using PMS as a function of ageing time.

of the AZO/PET films. In the visible region, the AZO film does not absorb, showing a slight reduction in transparency owing to interference effects produced by the difference in refractive index between AZO (\sim 2.0) and PET (\sim 1.58).

Figure 6 shows electrical resistivity as a function of ageing time for the samples under ambient conditions. According to some growth models³⁵, the film deposited for 5 min has smaller crystallites compared to the films deposited for 30 min. This greater number of the grain edges contributes to electron scattering, decreasing their mobility and consequently increasing resistivity. As for ageing, with a greater number of grain edges, there will be

a more pronounced incorporation of oxygen, increasing the oxidation rate and therefore the resistivity.

Both samples show an electrical resistivity immediately after deposition of less than $8.0 \ge 10^{-4} \Omega$ cm. Despite this, the thinnest film (50 nm), showed an increase in resistivity in a few days, stabilizing at t $1.3 \ge 10^{-2} \Omega$ cm. The film of 200 nm presented a slight increase, stabilizing at $1.2 \ge 10^{-3} \Omega$ cm, compatible with the value obtained for sample B2. This indicates that above a certain critical thickness the films are electrically stable. Below this value, the electrical properties suffer degradation, possibly because of the reaction of A1 atoms with oxygen that diffuses into the AZO film while the morphological structure is at the nucleation stage³⁶.

4. Conclusions

This study compared AZO/PET films deposited by *magnetron sputtering* at ambient temperature, using different plasma power supplies: RF (13.56 MHz) and pulsed at a frequency of 15 kHz (PMS). In addition, the formation of cracks was evaluated together with the electrical stability with ageing.

All of the AZO/PET samples present optical transmittances greater than 80% in the visible region. Morphological analysis revealed that all the films are polycrystalline and exhibit a wurtzite structure with preferential growth in the crystallographic direction of the (002) plane. The films produced using RF, however, show greater residual tensions, which contribute to the formation of cracks in the AZO film.

A greater density of cracks was observed for the AZO films deposited by PMS. Regarding the electrical resistivity, the AZO films synthesized by RF possess values above $1.0 \times 10^{-2} \Omega$ cm, while the films produced using PMS have values of about $1.0 \times 10^{-3} \Omega$ cm.

Finally, the film that gave the best performance was that deposited at a target-substrate distance of 4 cm, using the PMS source, for 30 min. Under this condition the film had a resistivity of $1.0 \times 10^{-3} \Omega$ cm, a transmittance of ~83%, presented no cracks and exhibited good stability in its properties upon ageing.

Therefore, PMS shows itself to be more adequate for the production of AZO films on PET, with potential application in the fabrication of cheap, flexible opto-electronic devices.

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