

# Oxide coatings on flexible substrates for electrochromic applications

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**Abstract.** One of the most studied classes of materials in the modern microelectronic devices are the metal oxides. There are different metal oxide films, such as electrodes, charge injecting and electrochromic coatings for displays or “smart” windows applications. This paper aims to describe the recent achievements for oxide coating deposition for flexible electrochromic displays. Although many deposition methods for production of such films have been developed, some of the achievements in the field of RF sputtering of transparent electrodes from indium-tin oxide on low-cost polyethyleneterephthalate substrate are presented. Attention is paid on some critical issues, such as films electro-optical parameters (sheet resistance, transparency in the visible range), adhesion, degradation due to stress and patterning ability.

## 1. Introduction

Interest in the electrochromic devices and particularly in the electrochromic displays has increased recently, because these types of displays have the ability to modulate the light in such way that effect of “ink on paper” appears, which is favourable for the eye perception. This effect is suitable for electronic readers, watches and electronic labels – applications, requiring high readability, high contrast, and wide viewing angle. Although the electrochromic display is non-emissive, it offers all these advantages in contrast to the liquid crystal displays (LCD) [1]. Because the e-readers or labels are portable, a long battery life is required and therefore low power consumption is needed for such applications. This is one of the reasons for the efforts to be directed to the electrochromic displays due to their valuable property - memory after the power is turned off, which additionally decreases the power consumption. As it is known the electrochromic layer changes its optical density or colour as a result of injection or extraction of ions and electrons, respectively from electrolyte material and from transparent conductors, causing redox reaction. The optical state can return back to initial condition if the voltage is reversed. The threshold electrical potential necessary for activation of the process is within the range of 1.5-4 V. Some of the most studied electrochromic materials are  $\text{WO}_3$  [2],  $\text{MoO}_3$  [3],  $\text{V}_2\text{O}_5$  [4],  $\text{Nb}_2\text{O}_5$  [5],  $\text{NiO}$  [6] and their blends [7,8]. To observe the image generated in the display structure, the electrode film should be highly conductive and transparent for the visible light. Typical materials, fulfilling these requirements are tin oxide ( $\text{SnO}$ ), indium tin oxide ( $\text{In}_2\text{O}_3:\text{Sn}$ ) and zinc oxide ( $\text{ZnO}$  – pure and doped by boron) [9,10]. As can be seen, the oxide coatings are one of the main components in the electrochromic devices. Flexible substrates are the basic for the next generation display panels, making them more compact, lighter, thinner, and giving the opportunity to be rolled

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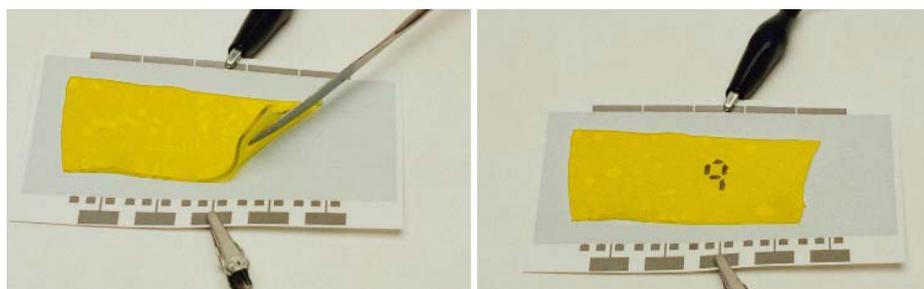
and brought in the pocket like a conventional sheets of paper [11]. However, there are still some challenges related to the technological development and some obstacles related to the mechanical durability during folding, that should be overcome. The main technological restriction is that the deposition process should be low temperature due to the lower melting point of the flexible substrates in comparison to the glass. From other side the process temperature is related to the film's microstructure and therefore to its electrophysical properties. Second aspect is the mechanical stress, induced in the films at bending, leading to their degradation and decreasing the long-term reliability of the device [12].

This paper aims to describe some of the latest materials and technologies for oxide film deposition on flexible substrates with its application in electrochromic devices. Results from vacuum sputtering of indium tin oxide as a transparent electrode on polyethylene terephthalate (PET) foil are presented more detailed. Most of the existing papers in the field present results for excellent devices performance and only mention that the substrate is flexible, but rarely can be found extended research about the impact of the internal stress induced in the films at flexing and how it affects to the performance. Here, some results concerning this problem are presented.

## 2. Oxide films for the electrochromic cells: materials and technologies for deposition

### 2.1. Transparent electrodes

These are the films, connecting the electrochromic display with the power supply. They should have a sheet resistance lower than  $30 \Omega/\text{sq}$  and transparency for the visible light higher than 88 % [13]. The most common methods for deposition of the thin electrode films are vacuum methods (electron beam evaporation and sputtering), chemical vapour deposition, spray pyrolysis and electrochemical deposition [14]. When the substrate is flexible special attention should be paid to the deposition temperature. One approach to improve the manufacturing efficiency and not involving high temperatures could be printing of conductive and polymeric inks on polyester substrate (figure 1). The origin of the deposited material is conductive metal oxide powder, dispersed in a polymer binder (for ITO it is styrene butadiene copolymer in toluene), ensuring durability at bending [15]. Electrochromic display with ITO replaced by antimony-doped tin oxide deposited onto  $\text{TiO}_2$  pigments was successfully produced for contrast white background electrode. Although the conductivity decreases in comparison with non doped ITO, the stability of the new coating towards aqueous electrolytes is better.



**Figure 1.** Seven segment printed electrochromic display [15].

Figure 1 is reprinted from *Solar Energy Materials & Solar Cells*, Vol.56, James P. Coleman, Puttanachetty Madhukar, John H. Wagenknecht. Printed, flexible electrochromic displays using interdigitated electrodes, Pages 395-418, Copyright (1999), with permission from Elsevier.

For flexible display roll-to-roll sputtering a system for room temperature deposition is specially designed to produce multilayer ITO/Ag/ITO electrodes on polyethersulfone substrates [16]. The silver film thickness is negligible (less than 15 nm) in comparison to the ITO for achieving the desired front panel electrode's parameters - sheet resistance  $4.28 \Omega/\text{square}$  and optical transmittance 89.28 %. In

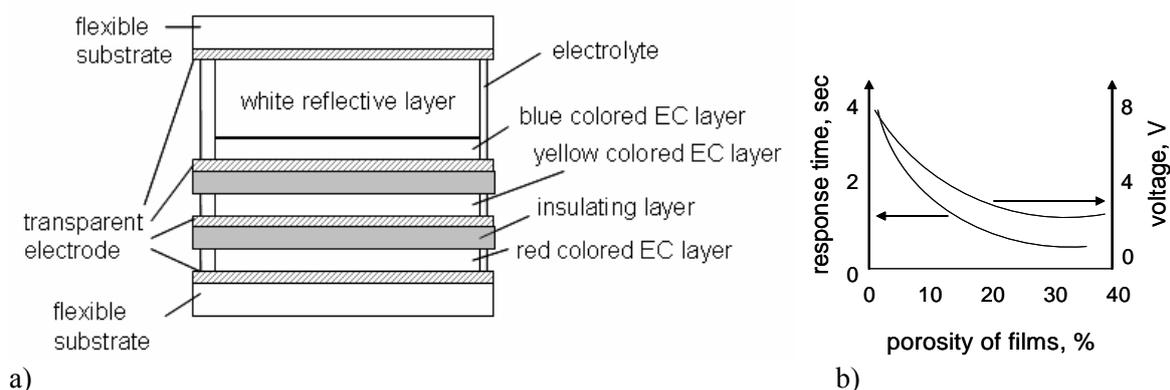
another research, RF and DC magnetron sputtering are applied respectively for ITO (50 nm) and copper (5 nm) deposition in ITO/Cu/ITO stack on polycarbonate substrate [17]. In this way, although the transparency is decreased (~70%) the resistance is extremely small ( $1.5 \cdot 10^{-4} \Omega \cdot \text{cm}$ ), which is a precondition for high charge injection efficiency. Multilayer structure indium zinc oxide/Ag(10nm)/indium zinc oxide(40nm)/Ag(10nm) is prepared on polyethylene terephthalate (PET) substrate by specially designed roll-to-roll DC sputtering system at room temperature and is analyzed by a bending system. This system provides bending with radius and frequency, respectively 10 mm and 60 Hz for 2 000 bending cycles [18]. Due to the silver nanoparticles existence the sheet resistance is ~5 ohm/square independently of the top indium zinc oxide film. Ag layer between both indium zinc oxide layers provides stable electrical conductivity even after the stress is applied due to the higher failure strain of the silver film. The deviation of the in-situ measured resistance, caused by the bending is less than 1 ohm/sq (20%). Aluminum-doped zinc oxide (AZO) thin films were studied as a potential candidate for flexible transparent conductive oxide polyethersulphone (PES) substrates by using an RF magnetron sputtering method at temperature below 200°C. The higher substrate temperature enhances the electric and the optical properties - the minimum electrical resistance for 250 nm thin film was  $2 \cdot 10^{-4} \Omega \cdot \text{cm}$ , and the optical transmittance was 90 % without any thermal and plasma damage to the PES substrate, which has higher degradation temperature than PET. [19]. However, the PES bending ability is weaker, due to its lower Young's modulus in comparison to the more popular PET and PEN.

Leterrier et al. observed the mechanism of revealing of cracks in pure (undoped) ITO thin films sputtered onto aromatic polyester. They found that the internal stresses were compressive, and that cracks in the ITO occur first around the pin-hole defects [20]. If the crack length is less than hundred times the thickness of the coating, it leads to increase in the electrical resistance with 10%. Otherwise, if the propagation of the crack is longer it leads to full failure of the flexible display. For evaluation of the fractional change in the resistance of ITO-coated by DC magnetron sputtering PET substrate as a function of the strain mechanical tests of the samples were made using tensile testing machine at constant crosshead speed [21]. Initial cracking occurs perpendicular to the loading direction in the ITO film at strain 2.75%. The number of cracks increases rapidly between 2.75% and 3.25% strain. At strain 6%, transverse cracking occurs due to lateral compression and at strain 10% cracks appear parallel to the primary cracks. The resistance increases sharply at threshold strain, depending on the ITO film thickness. An approach to improve the flexibility of conductive oxide electrodes on polymer substrates is introducing of surface roughness [22]. For this purpose regularly channels separated in equal distance were imprinted at elevated temperature onto Teflon® polymer surfaces. Amorphous and crystalline ITOs were magnetron sputtered. In this way ITO surfaces became unidirectionally wavy, which increased the surface area. As a result their bending stability was greatly improved - after 10,000 cycles at bend radius 10 mm, the resistance change was 50% of the conventional ITO.

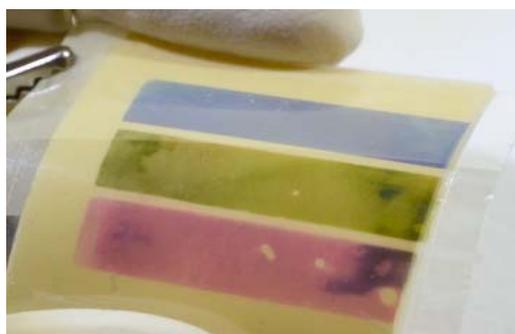
## 2.2. *Optically active materials*

The most common methods for deposition of the thin electrochromic films are the same as for the transparent electrodes - vacuum methods (electron beam evaporation and sputtering), as well as electrochemical deposition, chemical vapor deposition (CVD), electrochemical oxidation of tungsten metal [11]. Again the deposition temperature is crucial not only for preventing substrate from heat damages, but for the crystallinity of the film. For example amorphous and porous tungsten oxide can be produced at substrate temperatures lower than 100°C during e-beam evaporation. During sputtering, if the temperature is revealed to 200-300°C the  $\text{WO}_3$  films are dense and highly crystalline. Microstructure is tightly related to the electrochromic behaviour. Amorphous films have faster speed of switching between reduction and oxidation states, but for the crystalline ones the reaction is more stable and repeatable [23]. The same is valid for molybdenum oxide too, because its properties are very similar to  $\text{WO}_3$  [24]. Recently in the literature flexible designs of multicolour electrochromic display has been reported [25,26]. Nanostructured  $\text{TiO}_2$  layer is formed on PET by spin coating on the top of ITO electrode, using  $\text{TiO}_2$  water dispersions mixed with polymeric dies. New developed devices work stable even at bending radius less than 1 cm. The structure has several films colored in a

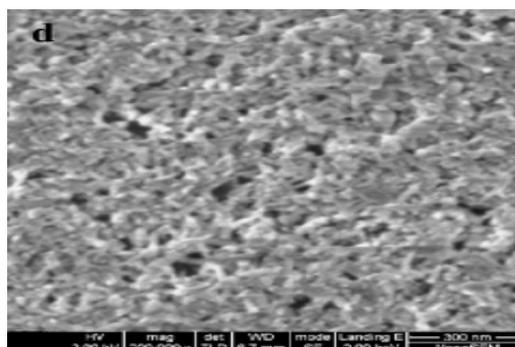
different way, separated by transparent electrodes (figure 2a). So here the porosity, due to low deposition temperature causes fluently ion migration, response time and voltage decrease (figure 2b). Photo of the fabricated samples can be seen in figure 3. Electrophoretic deposition process of  $WO_3$  nanoparticles has been reported in [28]. SEM image illustrates the porosity of the film, having high surface area (figure 4). Smaller than 100-150 nm nanoparticles facilitate faster ion-insertion ability, reduce the diffusion limited effects and improve transmittance modulation from 25 to 95 %.



**Figure 2.** a) Schematic of multilayer electrochromic display; b) Response speeds and activation voltage of multilayer electrochromic display with porous films [26].



**Figure 3.** Three colored electrochromic display on flexible substrate [27].



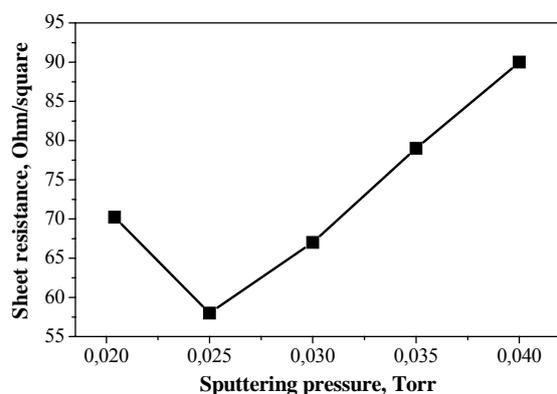
**Figure 4.** SEM image of a  $WO_3$  film deposited by electrophoresis on ITO/PET [28].

Figure 3 is reprinted from Y. Najjoh, T. Yashiro, S. Hirano, Y. Okada, S. Kim, K. Tsuji, H. Takahashi, K. Fujimura, H. Kondoh, Multi-Layered Electrochromic Display, ISSN-L 1883-2490/18/0375, ITE and SID, IDW '11, 375-378, Copyright 2011, with permission of Ricoh Globa; Figure 4 is reprinted from Thin Solid Films, Vol.512 /12, J. Bourée, Ch. M. White, D. T. Gillaspie, E. Whitney, S. H. Lee, A.C. Dillon, Flexible electrochromic devices based on crystalline  $WO_3$  nanostructures produced with hot-wire chemical vapor deposition, Pages 3596-3599, Copyright (2009), with permission from Elsevier.

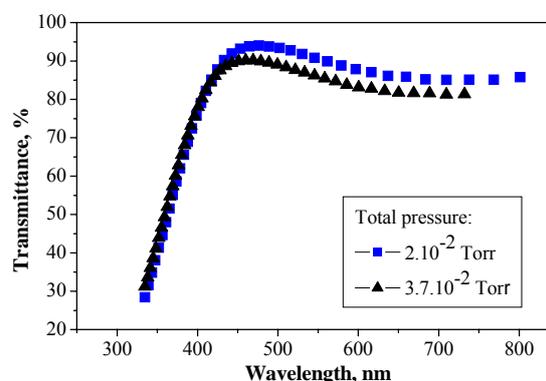
Report exists about growing of plasma assisted reactively evaporated  $WO_3$  on flexible Kapton substrate, withstanding at temperature during deposition of approximately  $200^\circ\text{C}$  [29]. At this temperature nanocrystalline  $WO_3$  films are produced. Samples show 73% optical transmittance in the visible range and coloration efficiency at a wavelength 550 nm of about  $45 \text{ C}/\text{cm}^2$ . Another example presents RF magnetron reactive sputtered  $WO_3$  on commercially available ITO coated polyethylenephthalate (PEN) substrate [30]. With decreasing plasma power and increasing total gas pressure, the crystalline phase disappear and the films is tend to be amorphous, which is the purpose, because such film exhibits better electrochromic properties.

### 3. Indium tin oxide films on PET deposited by RF sputtering –electro-optical properties and patterning ability

Although a variety of modifications has been developed in the materials and methods for adapting the transparent conductive coatings to the flexible substrates, indium tin oxide still takes one of the first places for display applications. As it can be seen from the previous section the electrode structure is rather complex in many cases (multilayers and doping) and sometimes flexibility is decreased due to using of less bendable, but thermally stable substrates. In the following text results are presented from simple, single layer deposition and tuning of the electro-optical properties of sputtered ITO films on PET by modification of the deposition regimes. PET pieces were exposed to UV light ( $\lambda=365$  nm and  $P=250$ W) for 5 minutes, leading to oxidation of the polymer backbone, adsorption of oxygen species and decreased roughness of the polymer surfaces [31, 32]. In this way surface energy of the already oxidized PET was increased, indium and tin particles can be easily bonded to the surface without need of high plasma power for insuring reaction kinetic for the initial ITO monolayers. This allows deposition at temperatures lower than 100-120°C, at which PET is less thermally sensitive. Sputtering pressure in the vacuum chamber should be also precisely tuned experimentally for ensuring suitable long free paths of the metal particles, according to the target-substrate distance and sticking them to the oxygen from the PET surface. There is a minimum in the sheet resistance for an argon concentration of 25 mTorr and then it gradually increases (figure 5). The reason is decreased sputtering throughput, due to the shorter mean free path of the particles and often collisions, leading to particles' energy losses. For the optimum sputtering pressure (related to the total pressure) the minimum achieved resistivity was  $\sim 57$   $\Omega$ /sq and the transparency - 87.8% (figure 6). As it can be seen from the XRD diffractogram of ITO (figure 7) its structure is mainly amorphous.

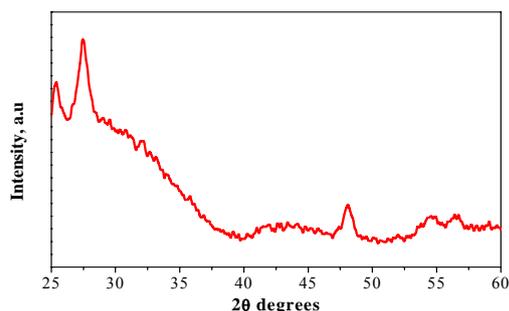


**Figure 5.** Sheet resistance versus sputtering pressure for ITO sputtered on PET substrate.

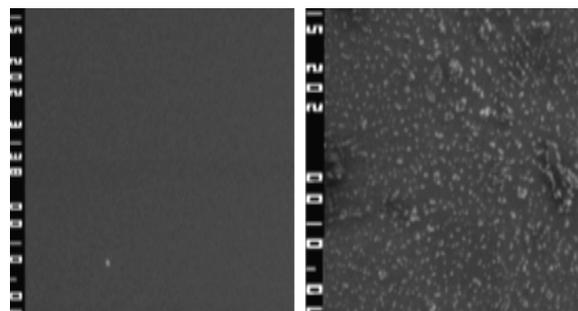


**Figure 6.** UV-VIS spectra of ITO coated PET at different sputtering pressures.

The strongest peak around  $2\theta = 30^\circ$  can be ascribed to orientation of the formed crystals preferentially in (222) direction and the peaks around  $45^\circ$  and  $55^\circ$  are due to PET substrate. ITO deposition by two stage change of the plasma power on PET foil is a new contribution in the described work. The influence of the plasma intensity on the ITO film's properties was established [33,34]. This modification of the deposition mode leads to changes in the way of arrangement of the microparticles. At the first deposition stage the sputtering voltage was set to 0.5 kV (or plasma power of 75 W), although the typical values for this parameter are in the range of 1.1-1.4 kV [35]. At the lower sputtering power, the temperature in the chamber was 62°C (more than 20°C lower than the substrate's melting point). 8 minutes after the lower temperature sputtering, plasma power was increased to 105 W, corresponding to deposition temperature of 75°C. It was established by SEM (figure 8) that the power increasing leads to reorganization of the ITO particles on the substrate surface and transition from amorphous to microcrystalline structure consisting of regularly distributed grains.



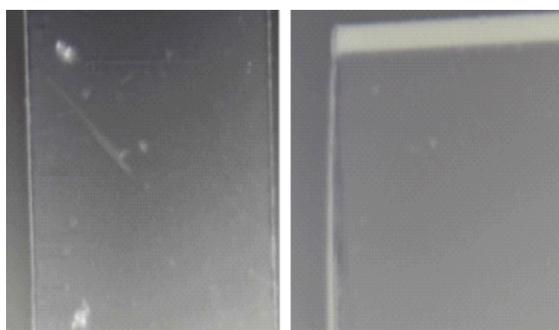
**Figure 7.** XRD spectrum of ITO on PET at the optimum sputtering pressure.



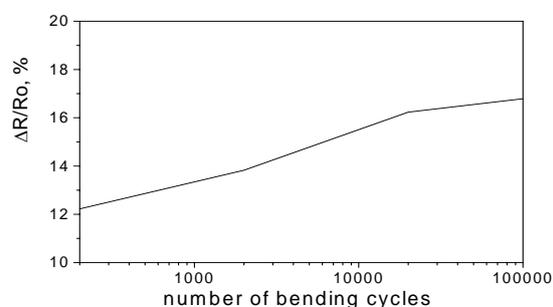
**Figure 8.** SEM of ITO: left – plasma power 75W; right – plasma power switched to 105W [34].

As can be seen from the left SEM image, the surface of the ITO film deposited at lower deposition temperature of 62°C is amorphous, but after plasma power increase and temperature rose to 75°C and crystallization mixed with amorphous underlayer phase presents. This structural change of the ITO film results in decreasing of the sheet resistance with 10 Ω/sq.

Two different rates of sputtering power change were set: 0.5 W/s and 3 W/s, but the power change rate of 0.5 W/s was selected as optimum, because ITO film adhesion to the PET substrate was found the strongest – at 70 N/cm<sup>2</sup> tensile force and peel angle of 90° the coating remained on the PET surface. At rates higher than 0.5 W/s the layer was separated from the substrate at 14 N/cm<sup>2</sup>, because the particles suddenly receive high kinetic energy and can not arrange by taking energetically favourable states on the surface. It results in irregularities, causing strain forces and weak adhesion. Optical microscopy (figure 9) shows ITO coated PETs after cyclic mechanical loading of 500 bends with curvature ~ 135° at different sputtering power change rates. The results show few micro cracks per area of 9 cm<sup>2</sup> for the highly adherent ITO film, produced at sputtering power change 0.5 W/s.



**Figure 9.** Optical microscopy images: left – rate of sputtering power change 3 W/s; right – rate of sputtering power change 0.5 W/s [34].



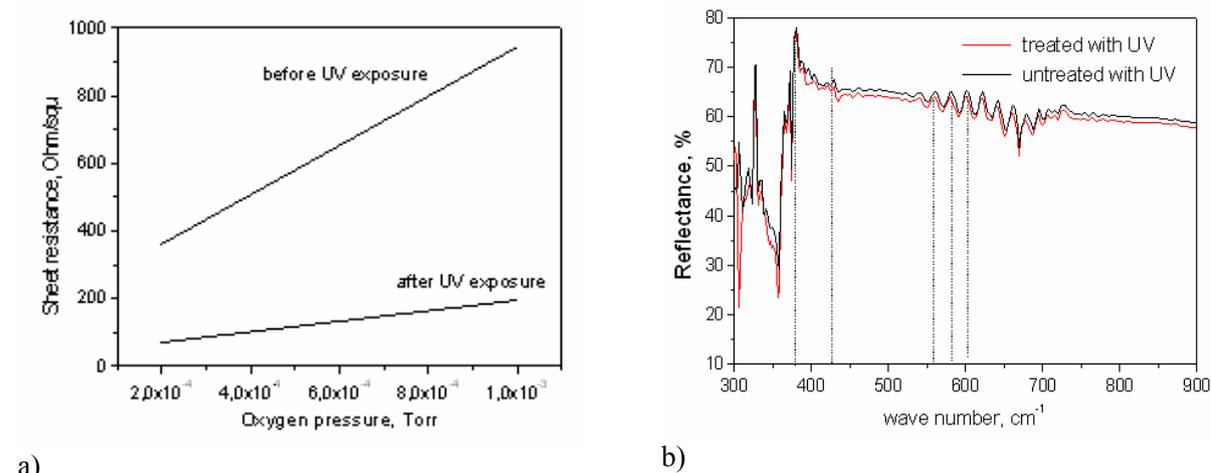
**Figure 10.** Relative change of the sheet resistance of sputtered ITO on PET after applying of dynamic mechanical load.

Figures 8 and 9 are reprinted from Int. J. of Reasoning-based Intelligent Systems, Vol.5, No.4, M. P. Aleksandrova; G. H. Dobrikov; K. A. Gesheva; G. K. Bodurov; I. N. Cholakova; G. D. Kolev, Deposition of transparent electrodes for the future generation of flexible displays, Pages 246 – 252, Copyright (2013), with permission from Inderscience Publishers.

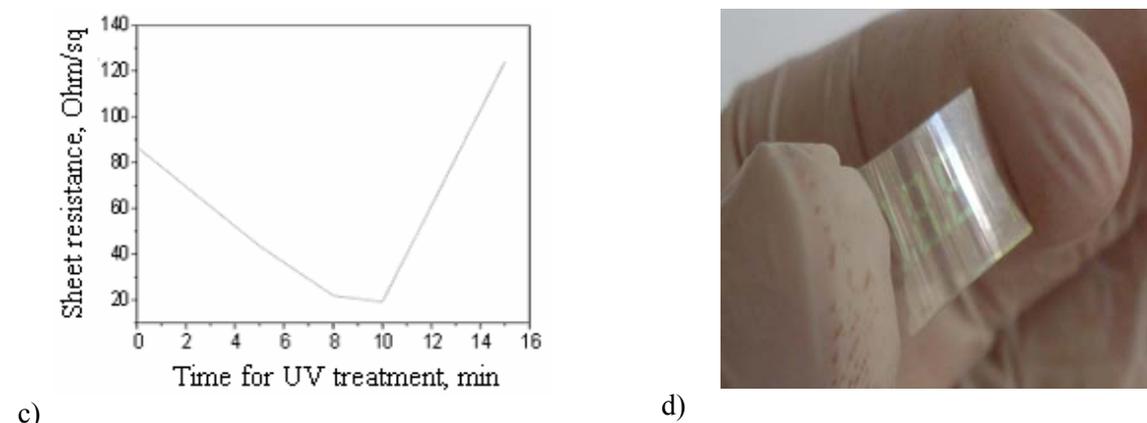
For estimation of the long term stability ITO coated PET was subjected to 100 000 numbers of mechanical bendings and the relative change of the ITO film resistance was measured. The result is illustrated in figure 10 and shows that the maximum deviation of the resistance  $\Delta R/R_0$  ( $R_0$  is initial value before bending) is 17 % at 100 000 repeated bending cycles and for 2 000 cycles it is 14%. For comparison in [18], the authors report about  $\Delta R/R_0 = 20\%$  at 2 000 cycles and additionally the electrode structure is multilayer.

Post deposition UV exposure with wavelength of 365 nm produced by mercury lamp and ozone formatting was conducted for ITO work function and surface energy increase [36], which results in

sheet resistance decrease (figure 11a). After 10 minutes of illumination the sheet resistance decreased to  $< 20 \Omega/\text{square}$  at the same oxygen pressure. These results demonstrate that after UV treatment it is possible to increase oxygen concentration in the chamber, making the metal oxide more transparent and still conductive. Typical FTIR spectra in reflectance mode, acquired in the range  $300\text{-}4000 \text{ cm}^{-1}$  for films deposited on PET at different post-deposition conditions are shown in figure 11b. The bonds strength was not affected by the UV exposure and no breakage or degradation was indicated. The peaks are situated at the same position, which is evidence for preserving the typical features of ITO film. Exposure causes crystallites formation, indicated by X-ray diffraction [37].



**Figure 11.** a) Sheet resistance of the thin ITO films at different oxygen pressures before and after 8 minutes UV exposure; b) FTIR spectra of the sputtered ITO layers on flexible PET substrate before and after UV treatment [34].



**Figure 11.** c) Sheet resistance of ITO sputtered on PET as a function of the UV illumination time [37]; d) ITO segments on PET, patterned by lift off process.

Figure 11c is reprinted from Journal of WASET, Vol.71, Aleksandrova, M. P.; Cholakova, I. N.; Bodurov, G. K.; Kolev, G. D.; Dobrikov, G. H., Influence of UV Treatment on the Electro-optical Properties of Indium Tin Oxide Films Used in Flexible Displays, Pages 1308-1311, Copyright (2012), with permission from WASET.

However, if certain critical UV dose is exceeded, degradation in the polymeric chemical bonds occurs and its resistance abruptly increases due to diffusion of released carbonyl groups into ITO film. At lower doses (shorter time than 10 minutes, 250W exposure intensity), the electrical behavior of the treated ITO layers can be ascribed to the activation of defects presenting in the disorder matter that

captures charge carriers. The trapped carriers are escaped after vibrational interaction with the electromagnetic wave and the sheet and specific resistance are decreasing (figure 11c). UV illumination should be taken into account during photolithographic patterning of ITO films. For PET/ITO patterning oxalic acid solution was used. It is isotropic etcher, so undercut effect is expected. For ITO thickness of 150 nm the bias was 6  $\mu\text{m}$  under both edges of the 2 mm wide strip shaped pattern – part of digital segment (figure 11 d) [38]. As alternative way for shaping of smaller patterns (such as pixels for example) lift off process is recommended, where there is no etching, so there is no bias effect.

#### 4. Conclusion

ITO films were deposited on flexible polymer substrate by developing a modified method of RF reactive sputtering at different plasma powers. It was found that plasma power affects the ITO films crystallization and adhesion strength. The substrates were treated by UV exposure before and after ITO deposition. It was found that it affects the degree of oxidation and activation of vacation states, which results in sheet resistance improvement. The minimum value achieved was 19.2  $\Omega/\text{sq.}$ , and the maximum transmittance of the visible light was above 87 %. In this way, a simple control of the electro-optical properties of sputtered ITO on PET without using of complex multilayered electrodes or doped materials is possible. The results are useful for further fabrication of flexible display.

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