Optoelectrical properties of PET spunbonded nonwovens deposited with ITO thin films by RF sputter coating

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Indium tin oxide (ITO) films were deposited on polyethylene terephthalate (PET) spunbonded nonwovens using radio frequency (RF) magnetron sputtering under different conditions. Indium tin oxide films with different thicknesses between 56 and 224 nm showed varying transmittances in the visible and ultraviolet regions. The transmittance of the nonwoven decreased as the thickness of the ITO film increased. The electrical properties of the material were also affected by the sputtering conditions. Larger sputtering power led to better electrical conductivity of the material. Higher sputtering pressure caused a decrease in electrical conductivity. The resistivity of the films first decreased with the increase in the temperature (until 150°C), and then increased as the temperature increased. The materials deposited with thicker ITO showed improved electrical conductivity.

Keywords: ITO, Nonwovens, Optoelectrical properties, RF sputtering, AFM

Introduction

Transparent conducting oxides (TCO) have attracted a lot of attention in recently years due to their great potential in many industries. The most widely used TCO is tin doped indium oxide (In$_2$O$_3$/Sn or ITO) which has a low electrical resistivity of down to almost 10$^{-4}$ Ω cm and high visible light transmittance of up to 80%.

Indium tin oxide (ITO) films have been increasingly used in a variety of applications, such as organic light emitting diodes (OLED), liquid crystals display (LCD), electromagnetic shielding, anti-ultraviolet, etc. In these applications, the device performance is strongly affected by the properties of the ITO.

Various techniques, such as vacuum deposition, chemical vapour deposition (CVD), sol-gel and sputter coating have been used to make ITO films on different substrates. Sputter coating with high depositing speed and excellent uniformity of nanofilms is one of the most commonly used techniques to deposit ITO films. Indium tin oxide films are usually deposited on glasses and some organic films.

In this article, ITO thin films were deposited on polyethylene terephthalate (PET) spunbonded nonwovens using radio frequency (RF) magnetron sputter coating. The optoelectrical properties of the PET nonwoven coated with ITO nanofilms were characterised by ultraviolet and visible light spectrophotometer (UV-vis) and four point probe method. An atomic force microscope (AFM) was also employed to characterise the microstructures of the films.

Experimental

Materials

The material used was PET spunbonded nonwoven with a mass of 100 g m$^{-2}$. Before depositing, PET spunbonded nonwovens were immersed in acetones for five minutes firstly, then washed by distilled water and finally dried in an electronic oven.

Sputter coatings

A magnetron sputter coating system JZCK-420B was used in this study. The nonwoven samples were put into an ultrahigh vacuum chamber with a base pressure of 5 × 10$^{-4}$ Pa. The ITO target used was a ceramic target of 2 inch with a composition of 97 wt-%In$_2$O$_3$ and 3 wt-%SnO$_2$. Pure Ar (99.99%) and O$_2$ (99.99%) were used as sputtering and reaction gases.

During the deposition, the gas flows of Ar and O$_2$ were controlled by mass flow valves. The thickness of the deposition layer was measured using a coating thickness detector (FTM-V) fixed in the sputtering chamber. The sample holder with a rotation speed of 100 rev min$^{-1}$ was introduced to improve the uniformity of ITO film deposited on PET fibres.

Optoelectrical properties

The optical properties of the PET nonwovens deposited with ITO thin films were analysed by ultraviolet and visible light spectrophotometer (UV-vis). The UV-vis spectroscopy used was a PerkinElmer Lambda 900 with a scanning accuracy of ±0.2 nm and the wavelength was chosen from 300 to 600 nm.
The resistivity was measured by the four point probe method (made by Baishen Technology). It is suited to measure resistance, electrical resistivity and square resistance. These four probes with $\phi 5$ mm are all made by Tungsten Carbide and the distance between each two probes is 1 mm. Samples were all tested for 20 times on the same direction and then the average values were obtained.

AFM imaging

The various microstructures of ITO thin films were analysed by an AFM CSPM 4000 (Benyuan Company Ltd). Scanning was carried out in contact mode AFM using a silicon cantilever. All images were obtained at ambient conditions. The scanning size was $3000 \times 3000$ nm.

Results and discussion

Optical properties of coated nonwovens

Indium tin oxide films with different thicknesses between 56 and 224 nm were prepared on PET spunbonded nonwovens. The uncoated sample shows the transmittance of about 70% in the range from 400 to 600 nm, indicating a good transmittance of visible light. The transmittance drops gradually from 70 to less than 5% in the range between 400 and 300 nm, indicating the UV shielding effect of the PET material. The ITO sputter coating significantly alters the optical properties of the PET nonwoven material, as displayed in Fig. 1.

Effect of coating thickness on optical properties

The transmittance of the PET sample decreases as the coating thickness is increased from 56 to 224 nm, as illustrated in Fig. 1a. The transmittance of the material drops from about 70 to 60% in the visible light range between 400 and 600 nm, as the ITO coating is 56 nm. The transmittance decreases gradually from 70 to less than 5% in the range between 400 and 300 nm, as the coating thickness is 56 nm. The transmittance is further reduced as the ITO coating is increased to 224 nm. The transmittance is reduced to $\sim 50\%$ in the wavelength between 400 and 600 nm, and the UV absorption is also improved as the coating thickness is increased to 224 nm. It is clearly indicated that the materials show almost the same transmittance when the wavelength reaches 300 nm. It is believed that the ITO films improve the light absorption and scattering and eliminate some reflection at the interface due to diffraction.5

Effect of oxygen flow on optical properties

Figure 1b shows the transmittances of the PET nonwoven with ITO films deposited at different flows of oxygen. The sample prepared with oxygen shows better transmittance, especially in the visible light between 400 and 600 nm. It is also observed that the increase in oxygen flow to 10 sccm can further improve the transmission of the material. This phenomenon is attributed to the oxidation process of the sputtered film during the sputtering.6

Electrical properties of coated nonwovens

Effect of sputtering power on resistivity

Figure 2 shows the change in the surface resistivity of the PET nonwoven deposited with ITO film at 50, 100, 150 and 200 W respectively. All samples were processed under a pressure of 0.5 Pa at room temperature, with an argon flowrate of 20 sccm without oxygen and a coating thickness of 100 nm. As the sputtering power increases, the surface resistivity is significantly lowered. The surface resistivity of ITO coated nonwoven decreases sharply in the sputtering power ranging from 50 to 150 W, and it tends to level off from 150 to 200 W. The ITO atoms which are bombarded per seconds by argon plasma are increased and the density of ITO atoms deposited on PET fibres is higher as the sputtering power is increased. Therefore, the surface conductivity of the material is improved. Considering the stability of sputtering deposited rate, 150 W was selected for the later experiments.
Effect of sputtering pressure on resistivity

This group of samples with a coating thickness of 100 nm was all deposited under the condition of 150 W, 20 sccm of Ar flowrate and room temperature. As shown in Fig. 3, the conductivity of the ITO coated nonwoven is improved with the decreased sputtering pressure. As the sputtering pressure increases, the number of ITO atoms arrived on the surface of the fibres decreases. Then free path of ITO atoms with electric is shortened and their kinetic energy is reduced, which directly causes fewer atoms deposited on the substrate. The growth of the sputtered particles is also limited, leading to the increased resistivity. The experiments indicate that the conductivity under 0.2 Pa is better than that under 0.5 Pa, however the crystallites of the former is much worse than the later, therefore the sputtering power of 0.5 Pa was selected.

Effect of temperature on resistivity

The electrical properties of the PET nonwoven deposited with ITO films sputtered under the selected power (150 W) and pressure (0.5 Pa) at various substrate temperatures of 50, 70, 100, 150 and 200°C are shown in Fig. 4. It is found that the resistivity decreases as the substrate temperature increases. The resistivity reaches a minimum value at the temperature of 150°C and then shows a little increase as the temperature is further increased to 200°C. This phenomenon shows a good agreement with the experiments of depositing ITO films on glass, which was investigated in the literature. It should be attributed to the change of carrier mobility and carrier density, which alters the crystallinity of the ITO films. The decrease in resistivity with the increase in the substrate temperature can also be explained by the fact that the grain size increases significantly with the increase in deposition temperature, thus reducing rain boundary scattering and increasing conductivity.

Effect of coating thickness on resistivity

Indium tin oxide films with thickness between 20 and 200 nm were prepared on PET spunbonded nonwovens. All were deposited under the same power (150 W), pressure (0.5 Pa), temperature (150°C) and Ar flowrate (20 sccm). The effect of the coating thickness on the surface resistivity is shown in Fig. 5. The surface resistivity of the PET nonwoven coated with an ITO film of 20 nm is as high as 136-2 kΩ cm. As the film thickness is increased to 100 nm, the resistivity rapidly decreases to 1.24 kΩ cm. Then the surface resistivity decreases slowly to ~1 kΩ cm as the coating thickness is further increased from 100 to 200 nm. This results are in good agreement with that reported by Dong-Ho Kim. Surface morphologies of ITO films with thickness of 100 and 200 nm are shown in Fig. 6. Tiny grains are found scattering on the surface of ITO films. The sputtered particles are clearly observed in the AFM image, and the average size of them is ~42 nm, which were analysed by the AFM software Image 4, as shown in Fig. 6a. The grains of ITO film with a thickness of 200 nm are also clearly visible and the uniformity looks improved. The average size of the grains is nearly 48.7 nm, as presented in Fig. 6b. Further studies on the effect of surface treatment on the crystallinity and electrical properties of the polymer substrate sputtered with ITO films are needed in the future work.

Conclusions

Indium tin oxide films with different thicknesses between 50 and 224 nm had various transmit values in the range 50–70%. The results show that the PET samples with ITO films on them had much lower ultraviolet (UV) transmittance than those without anything on. The transmission was decreasing with the increase in the thickness of the film.

On the other hand, ITO films brought better electric property to the PET sample, which was indicated by the experimental results. Lager sputtering power caused better electrical conductivity and higher sputtering pressure induced worse electrical conductivity. The
The resistivity of the films was first decreased with the increase in temperature (till 150°C), and then increased with increasing temperature. The thicker the films, the better the electrical conductivity is.

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