

Electrical and Mechanical Properties of Indium-tin-oxide Films Deposited on Polymer Substrate Using Organic Buffer Layer

Jeong In Han, *Member*, Chan Jae Lee, Sung Kyu Park, *Member*, Won Keun Kim and Min Gi Kwak

Abstract

The electrical and mechanical properties in indium-tin-oxide films deposited on polymer substrate were examined. The materials of substrates were polyethersulfone (PES) which have gas barrier layer and anti-glare coating for plastic-based devices. The experiments were performed by rf-magnetron sputtering using a special instrument and buffer layers. Therefore, we obtained a very flat polymer substrate deposited ITO film and investigated the effects of buffer layers, and the instrument. Moreover, the influences of an oxygen partial pressure and post-deposition annealing in ITO films deposited on polymer substrates were clarified. X-ray diffraction observation, measurement of electrical property, and optical microscope observation were performed for the investigation of micro-structure and electro-mechanical properties, and they indicated that as-deposited ITO thin films are amorphous and become quasi-crystalline after adjusting oxygen partial pressure and thermal annealing above 180 °C. As a result, we obtained 20-25 Ω /sq of ITO films with good transmittance (above 80 %) of oxygen contents with under 0.2 % and vacuum annealing. Furthermore, using organic buffer layer, we obtained ITO films which have a rather high electrical resistance (40-45 Ω /sq) but have improved optical (more than 85 %) and mechanical characteristics compared to the counterparts. Consequently, a prototype reflective color plastic film LCD was fabricated using the PES polymer substrates to confirm whether the ITO films could be realized in accordance with our experimental results.

Keywords : Indium tin oxide, polymer substrate, oxygen, annealing, organic buffer layer

1. Introduction

More attention is being given to plastic based devices because of their potential application as a lightweight, thin and flexible integrated circuit (IC) and flat panel display [1-3]. In recent years, portable information devices such as personnel digital assistant (PDA), mobile phone and smart card are increasing due to the development of computers and mobile communication systems. The demands now days are more compact, thin and lightweight inner device [4]. Therefore, many researchers have focused on the plastic based devices [5-

7]. For these applications, it is necessary to investigate the characteristics of the thin films deposited on polymer substrates. Indium-tin-oxide (ITO) films show a low electrical resistance and high transmittance in the visible range of an optical spectrum. Therefore, they play an important role as transparent electrodes for current electronic devices [8]. However, the resistivity of the ITO films on the polymer substrate is rather high to be adopted as transparent electrodes for an improved plastic-based device. It is a well known fact that the polymer substrates suffer mechanical and electrical changes at high temperatures applied during the heating process of the device fabrication [9-12]. Most of the changes are induced by a high thermal expansion and low thermal resistance in polymer substrates [13-15]. Using a novel device, stepped heating process and an organic buffer layer, we were able to eliminate the tensile force which induces the mechanical changes and thus,

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lessens the thermal expansion of the polymer substrates. In addition, we also report the properties of the ITO films deposited on the polymer substrates. There is few analytical reports on this subject, especially, on oxygen flow-out, annealing conditions, thermal expansion and gas absorption properties related to polymer substrates. We selected polyethersulfone (PES) as a substrate material because of its high optical quality (more than 90 % in optical transmittance), high thermal resistance (above / 190 °C) and compatibility with most semiconductor processing chemicals.

2. Experimental Procedures

2.1 Measurement of thermal strains for polymer substrates

In order to develop a new process that can diminish the thermal expansion in polymer substrates, we experimented the temperature dependence on thermal strains for various substrates at the heating slopes of 10 °C/s and 5 °C/s. The strains were obtained by measuring the changes of sample dilatation (z-axis) according to the substrate temperature. The data obtained in the experiments demonstrate the strain ratio between the sample length at room temperature and the elongated length at a specific temperature. The substrates used in the experiments were 180 μm-thick PES and Polycarbonate(PC) substrate supplied with 80 nm SiO_x as a gas barrier layer. The gas barrier layer was deposited on a bare polymer substrate by a rf-magnetron sputtering at a room temperature and subjected to annealing treatment.

2.2 Novel device and organic buffer layer to prevent thermal cracking in ITO films

As mentioned above, when a composite material consisting of a thin brittle film adhering to high-elongation substrates is subjected to a tensile force, multiple cracking emerge in the brittle film perpendicular to the direction of a stretching force [12-16]. In order to eliminate the tensile force, we invented a novel device and applied it to the deposition process. As shown in Fig. 1, pulling coils with the constant speed corresponding to the stretching force compensated for

the tensile force. In addition to the device, we used polyimide and acrylic material as an organic buffer layer for diminishing the tensile force. Therefore, we could obtain a flat polymer substrate without any cracking or defect in the ITO films. A rectangular substrate was cut from a roll of the film and loaded on the device. A thin aluminum (Al) plate uniformly heated by a heating coil was located just above the sample. The temperature of the Al plate was monitored and feedbacked to the input power of the heater so that the plate was kept at a prescribed temperature. In addition, water with the constant temperature was circulated in the wall of the chamber to keep it at a fixed temperature. In this manner, uniform heating in the chamber and deposition process at a low temperature was realized though ambient temperature of the sample position was slightly different from the temperature of the Al plate. This temperature - controlled vacuum chamber was also used in a vacuum annealing process.

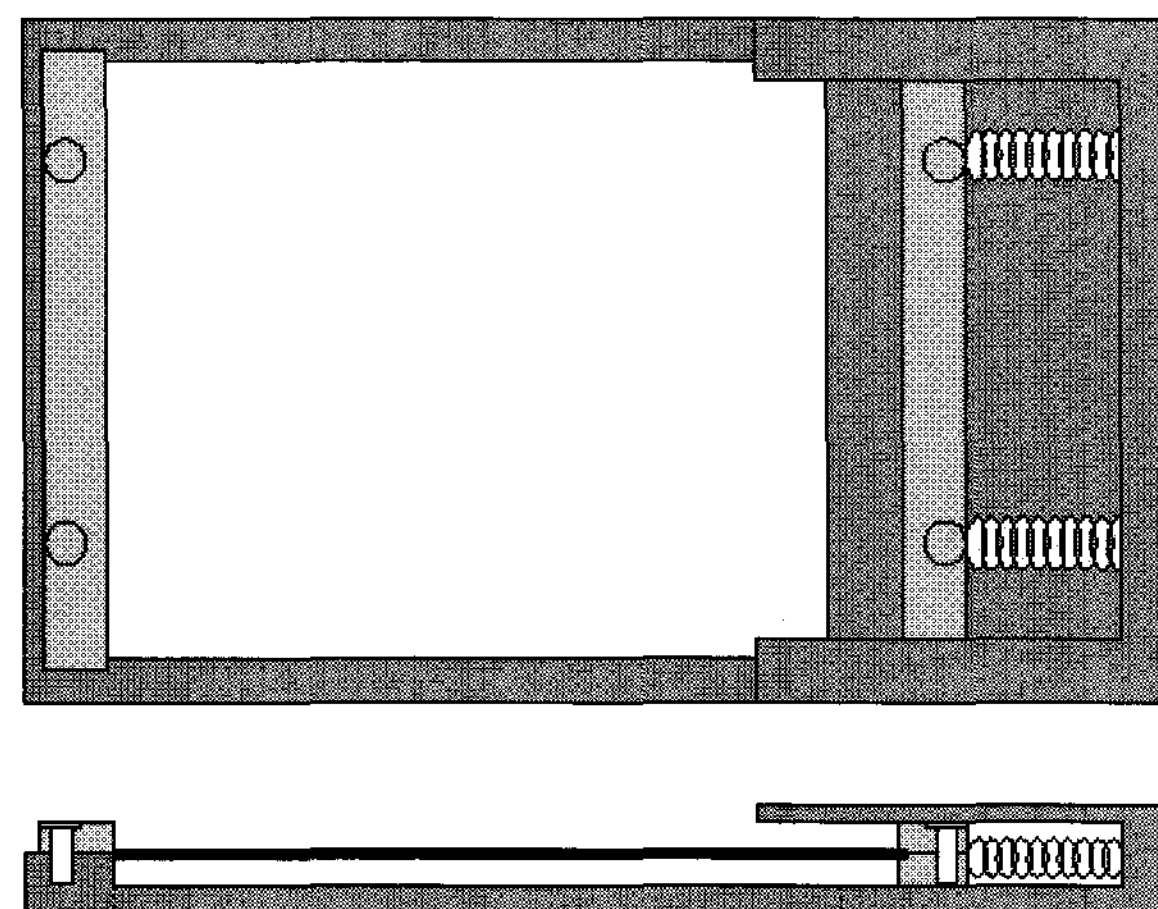


Fig. 1. Schematic view of the novel device to elimin.

2.3 Deposition of ITO films on polymer substrates

Deposition of ITO films on polymer substrates was performed by the rf-magnetron sputtering system which was modified for this study. The ITO target used for these experiments was a hot pressed In₂O₃ containing 10 wt% SnO₂. The material and size of the substrate were PES and 85 × 80 mm², respectively. The distance between the target and substrates was about 60 mm. The deposition process was preceded in the mixture atmosphere of argon and oxygen gases where the gases were controlled by a mass flow meter. Ar/O₂ was controlled in the range from 0.2 % to 15 %. The

minimum O₂ content is 0.2 %, which is the limit to be able to be controlled by our system. Base pressure of the sputtering system was 1.6×10^{-6} torr, the process pressure was 3.2×10^{-3} torr and the sputtering power applied in the process was 136 W. To control the electrical resistance and optical transmittance of the ITO films, various mixtures of the argon and oxygen were used as sputtering gases. The film thickness was controlled by adjusting the deposition time to 1000 ± 100 Å. The deposition process was also accomplished at a relatively low temperature to reduce the thermal expansion of the polymer substrates and annealing experiments were carried out using a conventional oven that has an air circulation facility or in the sputter chamber itself. Although the sputtering process was accomplished without an additional substrate heating, the temperatures of the chamber rose up to 64–78 °C due to the plasma process. The substrate was annealed for 1 hour since its surface reached the set temperature. Moreover, in order to diminish the thermal expansion of the substrates, the temperature was increased slowly by a stepped heating process. Finally, optical, electrical and chemical properties of the ITO films deposited on the polymer substrates were investigated for the application of the plastic-based devices.

3. Results and Discussion

3.1 Analysis of polymer substrates

In contrast to glass substrates, polymer substrates have a low thermal resistance, non-rigidness, weak mechanical characteristics and a high thermal expansion coefficient. The values of 44 ppm/K for PES and 37 ppm/K for PC are one order of magnitude higher than those of glass or ITO films [17]. The difference in thermal expansion between polymer substrates and ITO films can cause serious problems which will reduce the conducting of degrade the conductive property of the ITO films. As temperature increases, polymer substrates stretch in all directions at the rate of 37–44 ppm/K, but the ITO films deposited on the substrates does not stretch at the same rate. This large thermal expansion mismatch can produce significant thermal stress upon heating, leading to cracking and delamination of the thin films during subsequent processing. As shown in Fig. 2, it is

evident that glass substrates show strain of one order of magnitude lower than that of polymer substrates. It should also be noted in Figure 2 that lower heating slopes reduce the thermal strains remarkably. It is considered that these decreases of thermal strain can reduce the mismatch of thermal expansion according to Hooke's law and Poisson ratio (generally referred to 0.4 – 0.5 in polymer materials) and accordingly lessen the stress in the ITO films, which in turn result in a lower chance of defect during the sputtering process. Another distinct property of polymer substrates from glass substrates is in gas and moisture absorption of the substrates, which causes degradation of the ITO films and prevents their good adhesiveness on the substrates. Therefore, most polymer substrates used for plastic-based device require a gas barrier layer and special treatment. Nevertheless, they still show relative high absorption rate compared to the glass substrates. The O₂ and H₂O vapor permeations of this barrier layer are known to be 0.2 cc/m² · day · atm and 5 g/m² · day, respectively [18]. The substrate used in the experiment was 180 µm-thick PES supplied with 80 nm SiO_x as a gas barrier layer. Polyethylene terephthalate (PET) also have widespread availability, however its maximum use temperature of 120 °C and a high birefringence severely constrain the processing environment in our case. At sustained temperature of above 120 °C, PET undergoes substantial shrinkage, as well as a decrease in optical transparency and flexibility. Therefore, the experiment on PET material will need to be done in latter experiments.

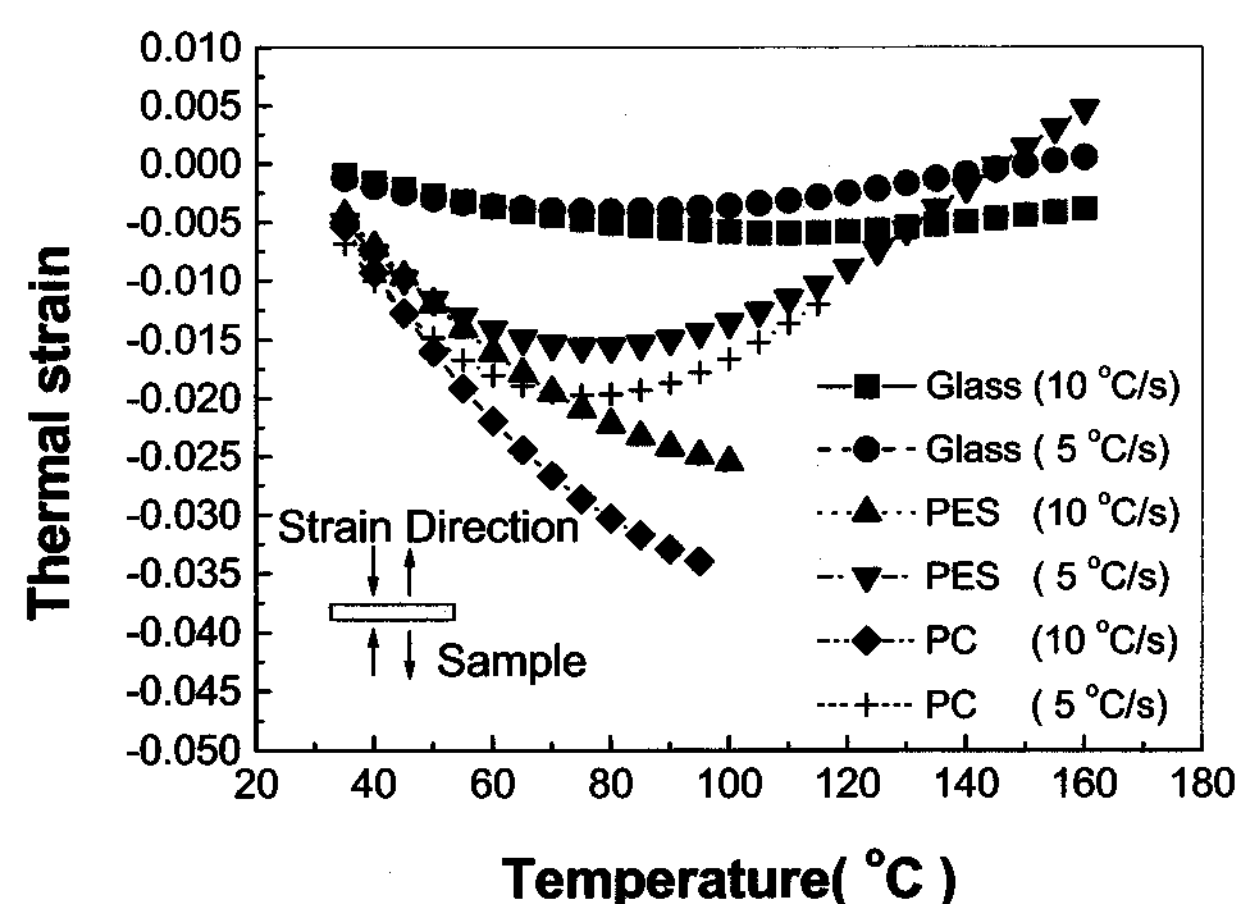


Fig. 2. Variations of the thermal strains for heating slopes.

TABLE 1. Deposition parameters, sheet resistance and wavelength at 40 % transmittance of the selected samples (Total gas pressure: 3.2×10^{-3} Torr, Power : 136W, Substrate : PES.

Sample	O ₂ partial pressure (%)	DC bias (V)	Dep. time	Sheet resistance	Wavelength at 40% transmittance	Film thickness
1	0.2	76-80	18 min.	25-28 Ω/sq.	385 nm	1030 Å
2	0.7	77-79	18	58	380 nm	1010
3	1.5	77-79	20	1K	370 nm	970
4	7	76-77	25	7.3K	362 nm	980
5	12	76-77	28	70K	355 nm	960
6	15	77-78	28	180K	350 nm	940

3.2 Influence of oxygen partial pressure during film deposition

Previously, it has been reported that oxygen partial pressure affects the electrical resistance, optical transmittance and deposition rate [19-20]. The dependence of the properties on oxygen partial pressure has been explained on the ground of oxygen vacancy, film structure theory and oxidation phenomenon. In this study, oxygen partial pressure varied from the minimum (0.2 %) possible to control by the mass flow meter up to one level (15 %). In Table 1, an overview of the samples investigated in this section is given, and Fig. 3 shows the influence of oxygen partial pressure on the electrical sheet resistance of the ITO films deposited at room temperature. The ITO films deposited under 0.6 % and 0.2 % oxygen partial pressure on both glass substrates and polymer substrates showed minimum values of 15 Ω/sq. and 25 Ω/sq., respectively, which were measured using a standard four-point probe technique. The sheet resistance increased with increasing oxygen partial pressure and the relation between sheet resistance and

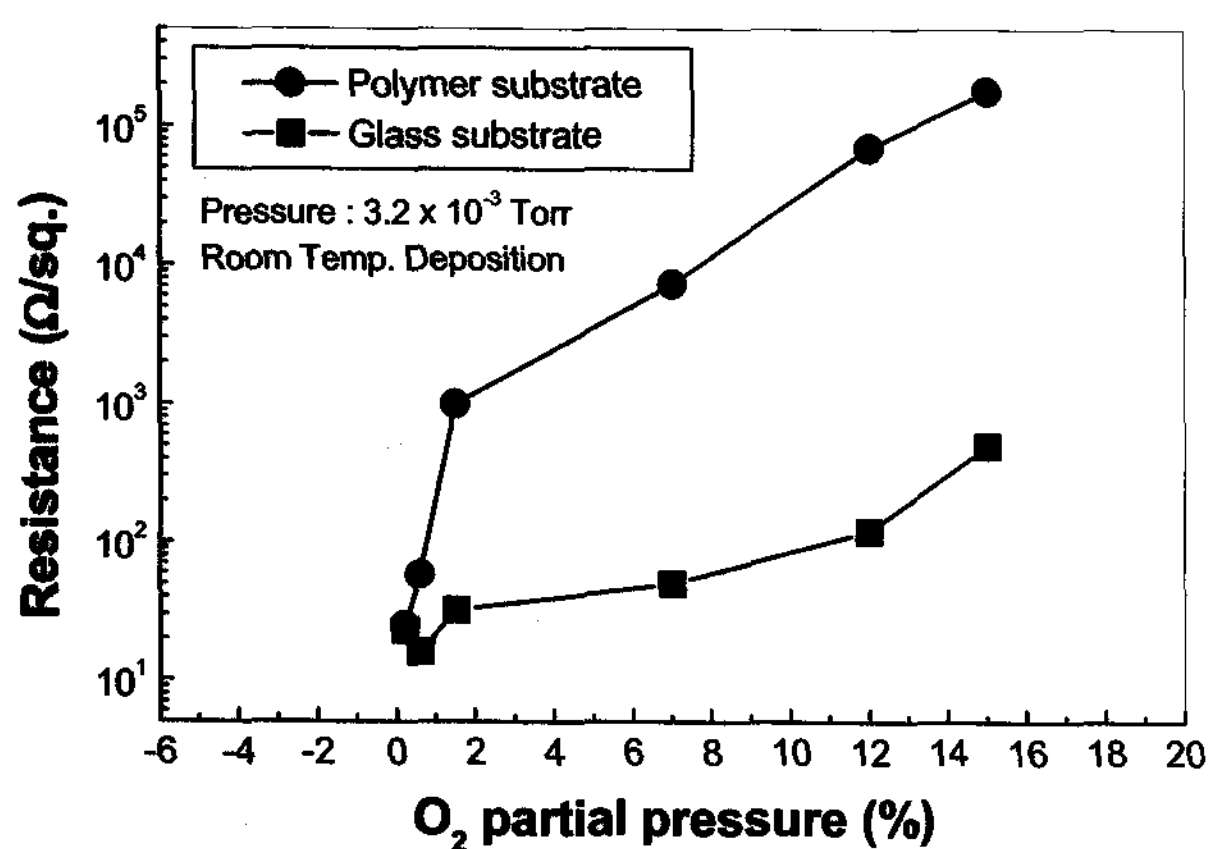


Fig. 3. Influence of the oxygen partial pressure on the sheet resistance.

the oxygen partial pressure is shown in Fig. 3. The existence of the minimum resistance is a well-known behavior of ITO films. It is supposed that the carrier density and mobility is determined by oxygen vacancies and grain size, respectively. On the bases of the assumption, we expect that higher carrier densities can be realized in the state of relatively low oxygen partial pressure and vice versa, In addition, higher mobility can be realized at higher oxygen partial pressure. As the two mechanisms cause opposite effects, the specific resistance shows a minimum value at a certain oxygen partial pressure. Though the same mechanism can be applied to the polymer substrates, the partial pressure to yield the minimum sheet resistance is relatively low compared to that of the glass substrates. It is considered that the difference of optimum oxygen partial pressure for the minimum sheet resistance should be due to the oxygen gas and moisture absorption of the polymer substrates. The polymer substrates have a tendency of absorbing oxygen gas and moisture much more than glass substrates. Therefore, the polymer substrates have high oxygen contents in the structure of polymer matrix and thus the oxygen may flow out resulting in increase in oxygen partial pressure in the deposition process. Fig. 4 shows XRD peaks of the ITO films deposited on the polymer substrates and glass substrates under 0.2% oxygen partial pressure. The main peaks of the ITO films appeared at (222) and (400) but those of the ITO films on glass substrates showed lower intensity. Other results reported on the XRD analyses of ITO films indicated that the intensity of (222) and (400) peaks increased with a rise of oxygen partial pressure [21]. Therefore, it is evident that the residual oxygen included in the polymer structure brought about the difference in optimum

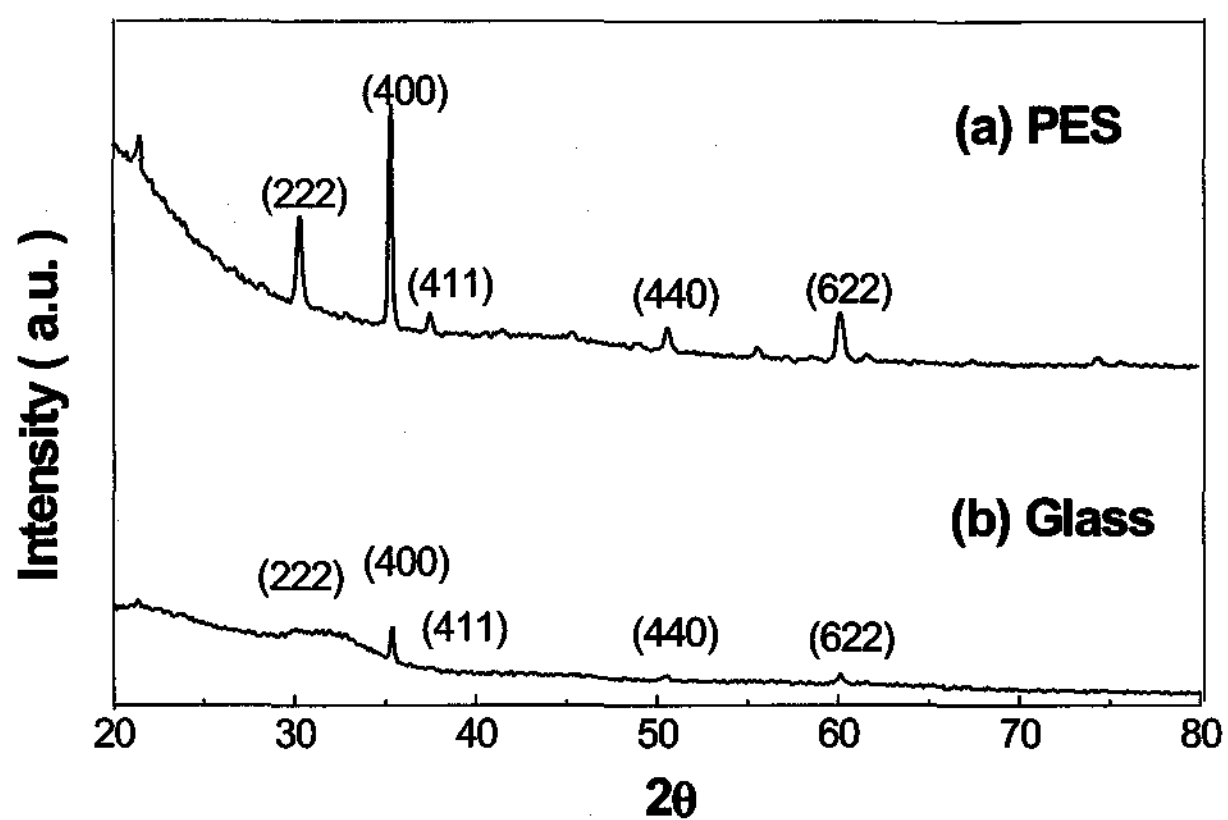


Fig. 4. XRD data of the ITO films deposited under the room temperature at 0.2% oxygen partial pressure for (a) PES substrate and (b) glass substrate.

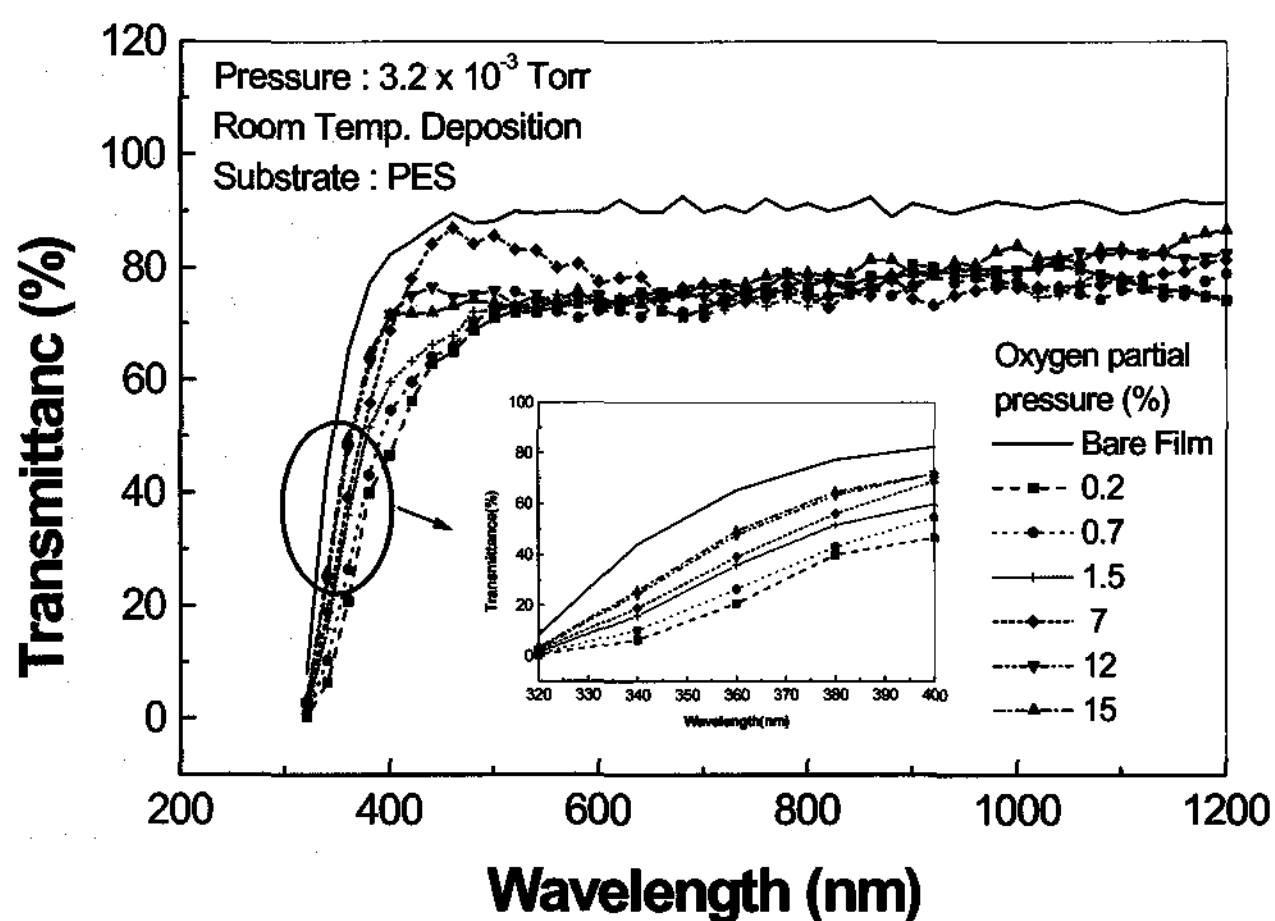


Fig. 5. Optical transmittance of the ITO films deposited on the polymer substrates as an oxygen partial pressure.

oxygen partial pressure for the minimum sheet resistance. Fig. 5 shows the optical transmittance of the ITO films deposited on polymer substrates as a function of the oxygen partial pressure. The transmittance spectrum of the ITO films was measured in the range of 320 nm and 1200 nm wavelengths. Because the reference material used in the measurement was air, the transmittance data depicted in Fig. 5 show the transmittance of ITO films including a bare substrate. The transmittance of the bare substrate was also measured for comparison. As shown in Fig. 5, the bare substrate has a transmittance of above 90 % almost in the visible ranges and ITO films also show a transmittance of 75~80 % in the range of 450-800 nm. It is a well-known fact that an optical transmittance has a direct relation with the band gap of ITO films. Generally, the band gap of ITO films is greater than 3.75 eV although a wide range of values from 3.5 to 4.5 eV have also been reported in the

literature [21,22] The fundamental absorption edge which lies near the ultraviolet region of the visible spectrum shifts to the shorter wavelength as carrier concentration decreases. This is in accordance with the Moss-Bernstein shift which describes an $N^{2/3}$ dependence of the band gap. From this experiment, it was found that the increase of oxygen partial pressure induces the decrease of carrier concentration, resulting in the increase in transmittance and decrease in sheet resistance. In addition, as shown in the small box in the Fig 5., the increase in the transmittance with the increase in oxygen partial pressure is revealed remarkably in the short wavelength range.

3.3 Influence of post-deposition annealing

Sheet resistance, optical transmittance and etching property were measured before and after annealing under an air atmosphere and a vacuum atmosphere, respectively. There were some differences in electrical and optical properties between the air annealing and the vacuum annealing. Fig. 6 shows the dependence of the sheet resistance on the annealing atmosphere and temperature. The samples were deposited under a room temperature and then the sheet resistance was measured by a standard four-point probe technique. The initial value of not-annealed sample was 25 Ω /sq. In the vacuum atmosphere, the sheet resistance decreased by 10 % with an increase of annealing temperature. It is known that the decrease of sheet resistance according to the increase of the temperature is attributed to the grain growth or the crystallinity of ITO films [23]. However, in the air atmosphere, there was little difference of sheet

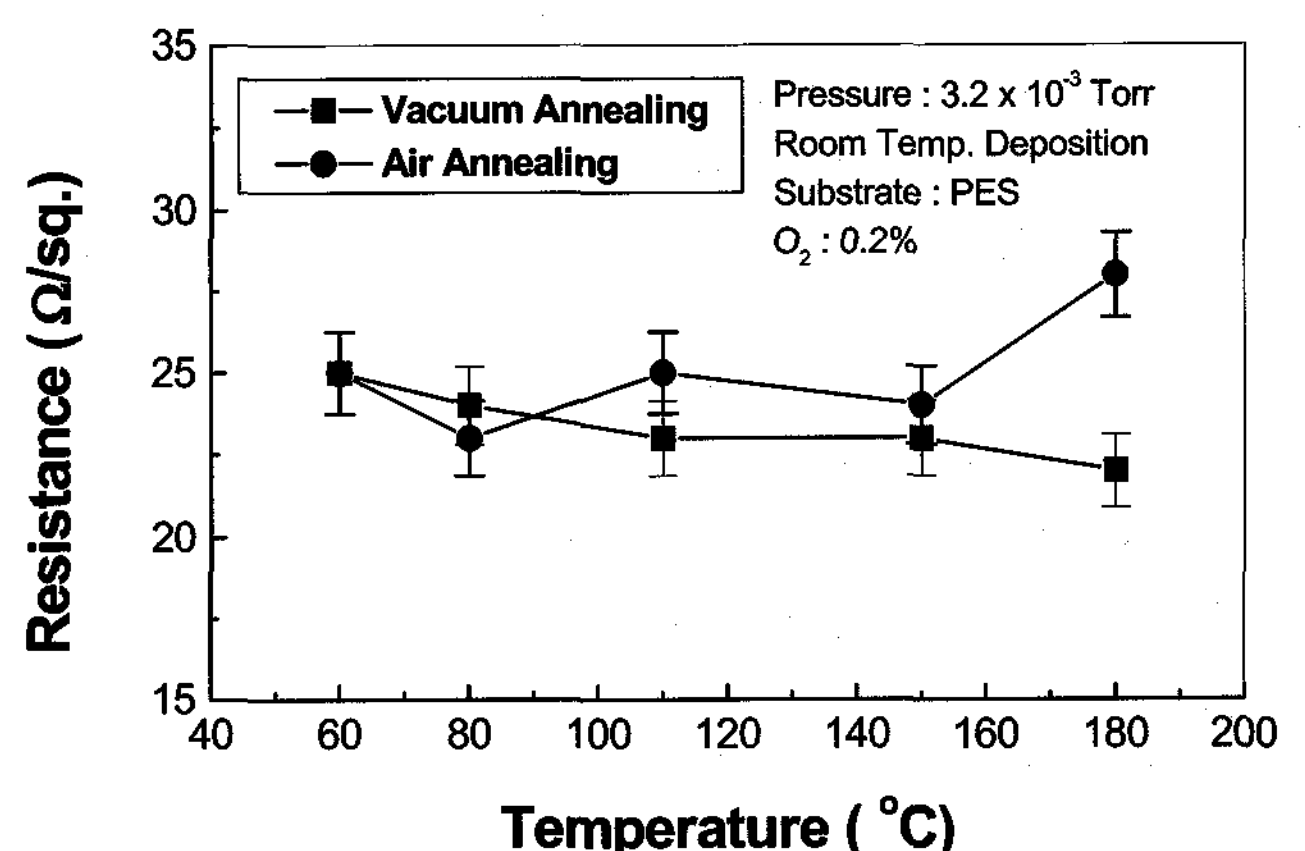


Fig. 6. Influence of the annealing temperature and atmosphere on the sheet resistance of the ITO films deposited on the polymer substrates.

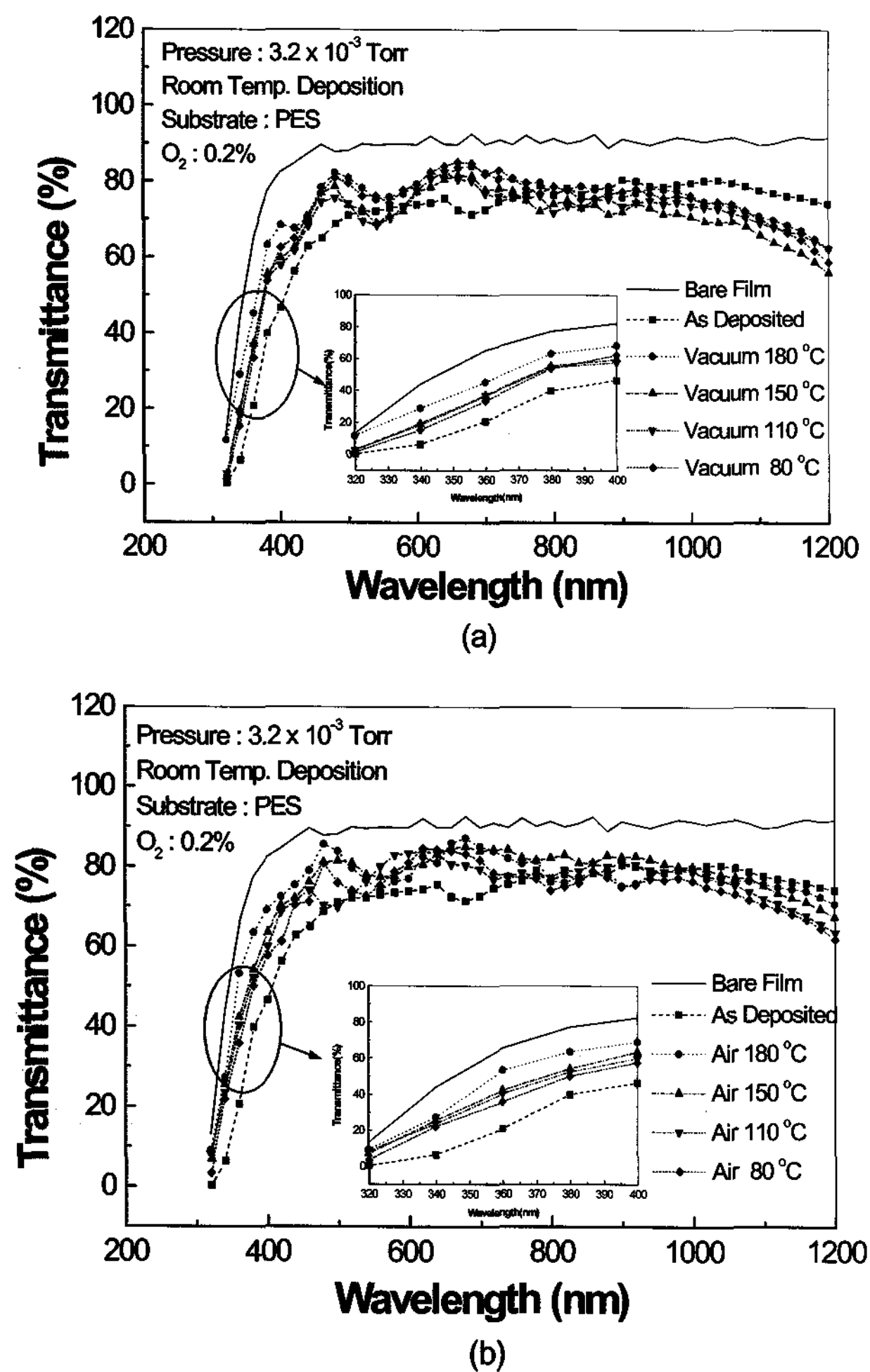


Fig. 7. Variations of the optical transmittance in the ITO films deposited on the polymer substrates before and after annealing at, (a) a vacuum atmosphere and (b) an air atmosphere.

resistance compared with the initial value except specimens annealed at 80 °C and 180 °C. Maximum sheet resistance was achieved at 180 °C, and distinctive trend in the sheet resistance on annealing temperature was not observed in the data. The main difference in the vacuum annealing and the air annealing is in the existence of oxygen. According to other literatures [8, 23], the oxygen diffusion from an air atmosphere into ITO films may occur at the temperature of higher than 200–300 °C. One of its effects is that it increases sheet resistance and decreases transmittance by the oxygen vacancy theory described in the previous section. However, because the polymer substrates used for these experiments have low thermal resistance, a maximum temperature to increase was limited to 180 °C. Following the oxygen diffusion theory on the temperature, it is appropriate to say that no evident changes would be found at such a low temperature in the case of the air annealing treatment. In

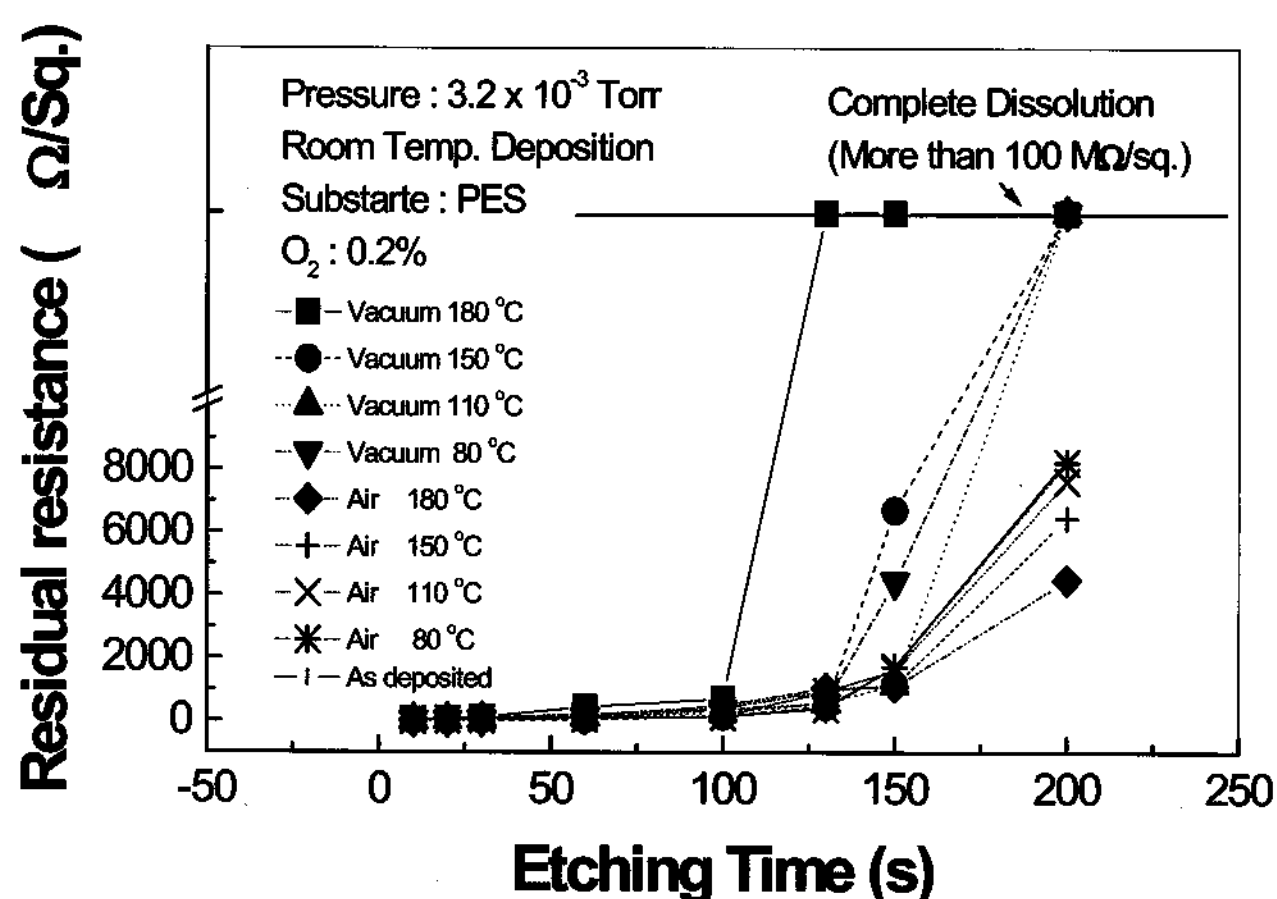
contrast to glass substrates, polymer substrates have more oxygen content inside the structure. Therefore, it is considered that the small increase in sheet resistance at a temperature of 180 °C is attributed to the oxygen diffusion in spite of negligible quantity. The same theory can be applied to the vacuum annealing, however, we think crystallinity or grain growth is a dominant factor in the vacuum atmosphere. Vacuum annealing at such a relatively low temperature may accelerate little crystallinity or grain growth process of the ITO films resulting in the decrease in sheet resistance, as shown in Fig. 6. Fig. 7 shows the transmittance of ITO films before and after the annealing process. In Fig. 7, it could be found that both the two annealing play a role of the improvement of the transmittance. The improvement could be explained on the aspects of little crystallinity and oxygen diffusion processes. Higher transmittance was obtained under the air atmosphere than under the vacuum atmosphere. Therefore, it is considered that the higher transmittance obtained under the air atmosphere was resulted from co-action of both the two processes. On the other hand, under the vacuum atmosphere, only little crystallinity contributed to the transmittance because the vacuum atmosphere has no oxygen gas. It has been reported that film crystallinity also affect the band gap of ITO films, similar to the oxygen effect. Moreover, a scattering effect related to the grain growth should not be ignored. Apart from the atmosphere, the increase in the annealing temperature increases the grain size, which results in the film structure to have high quality and density. The increase in grain size diminishes optical scattering and consequently increases transmittance. All these factors affect the transmittance of the ITO films, especially at a short wavelength range. Fig. 8 shows the dependence of etching rate on the annealing atmosphere and temperature. In the plastic-based devices manufacturing, an etching property is an important factor because it is one of the critical factors to identify electrode pitch and width which contributed to the resolution and device size. The major requirement of ITO films for high-pitch and high-resolution device is an efficient etching property under wet etching conditions, which does not bring about any short or open circuit in the electrode patterns. As shown in Fig. 8, the increase in temperature under vacuum atmosphere improved etching property. The time for complete etching of the ITO films annealed under the air atmosphere was twice as long as

TABLE 2. Annealing parameters, sheet resistance and wavelength at 40 % transmittance of the selected samples (Total gas pressure: 3.2×10^{-3} Torr, Power : 136W, Oxygen partial pressure : 0.2 %, Substrate : PES).

Sample	Dep. time	Annealing	Sheet resistance	Wavelength at 40% transmittance	Film thickness
1	18 min.	180 °C (Vacuum)	22 Ω /sq.	355 nm	1030 Å
2	18	150 °C (Vacuum)	23	362 nm	1020
3	18	120 °C (Vacuum)	23	362 nm	1040
4	18	80 °C (Vacuum)	24	365 nm	1030
5	18	180 °C (Air)	25	349 nm	1020
6	18	150 °C (Air)	28	356 nm	1030
7	18	120 °C (Air)	24	358 nm	1030
8	18	80 °C (Air)	25	365 nm	1050

TABLE 3. Sheet resistance of the ITO films deposited on glass substrate and polymer substrate treated with buffer layer.

Glass / ITO	15-20 Ω /sq
Glass / Buffer / ITO	27-30 Ω /sq
PES / ITO	25-28 Ω /sq
PES / Buffer / ITO	40-45 Ω /sq

**Fig. 8.** Variations of the residual resistances measured after the etching process with the annealing atmospheres and temperatures.

air atmosphere was twice as long as that of the ITO films annealed under the vacuum atmosphere. It is considered that the poor etching property of the ITO films annealed under the air atmosphere was a result of the impurities diffused by oxygen. From these experiments, it is evident that a little crystallinity and oxygen diffusion also play a major role in the etching property of the ITO films. However, the detailed mechanism of both the two processes should be further investigated.

3.4 Influence of organic buffer layer

In order to improve the mechanical property of the ITO thin films, we used an organic material as a buffer layer. The materials used for the layer were polyimide and acrylic polymer for the enhancement of affinity to the polymer substrates. Therefore, we obtained a rather flat substrate deposition of ITO films with good adhesion and without any cracking, as shown in Fig. 9 and Fig. 10. Moreover, this layer improved the transmittance of ITO thin films, which might be the result of oxygen contents included in the layer. Figure 11 shows the transmittance of the various substrates, and it indicates that the organic buffer layer improves the transmittance in all of the visible ranges. Table 3. shows the sheet resistance of the ITO films deposited on various conditions. As shown Table 3, the buffer layer also affect the sheet resistance of the ITO films. According to the experimental results, it can be concluded that the polymer matrix of the buffer layer lessened the stress between the polymer substrate and the ITO films added to improve the transmittance resulting from the oxygen contents added in the matrix structure. However, the detail investigations on the complex mechanism of the polymer matrix and the oxygen contents are needed in future works.

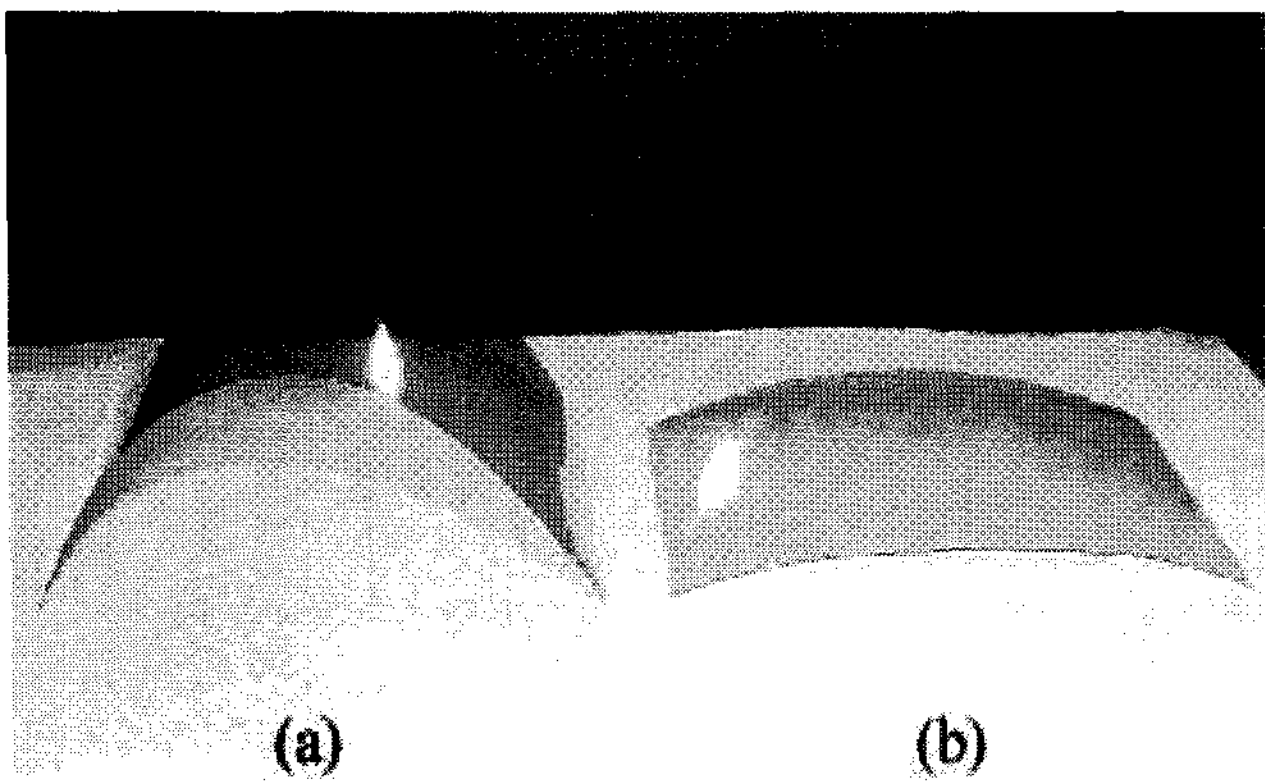
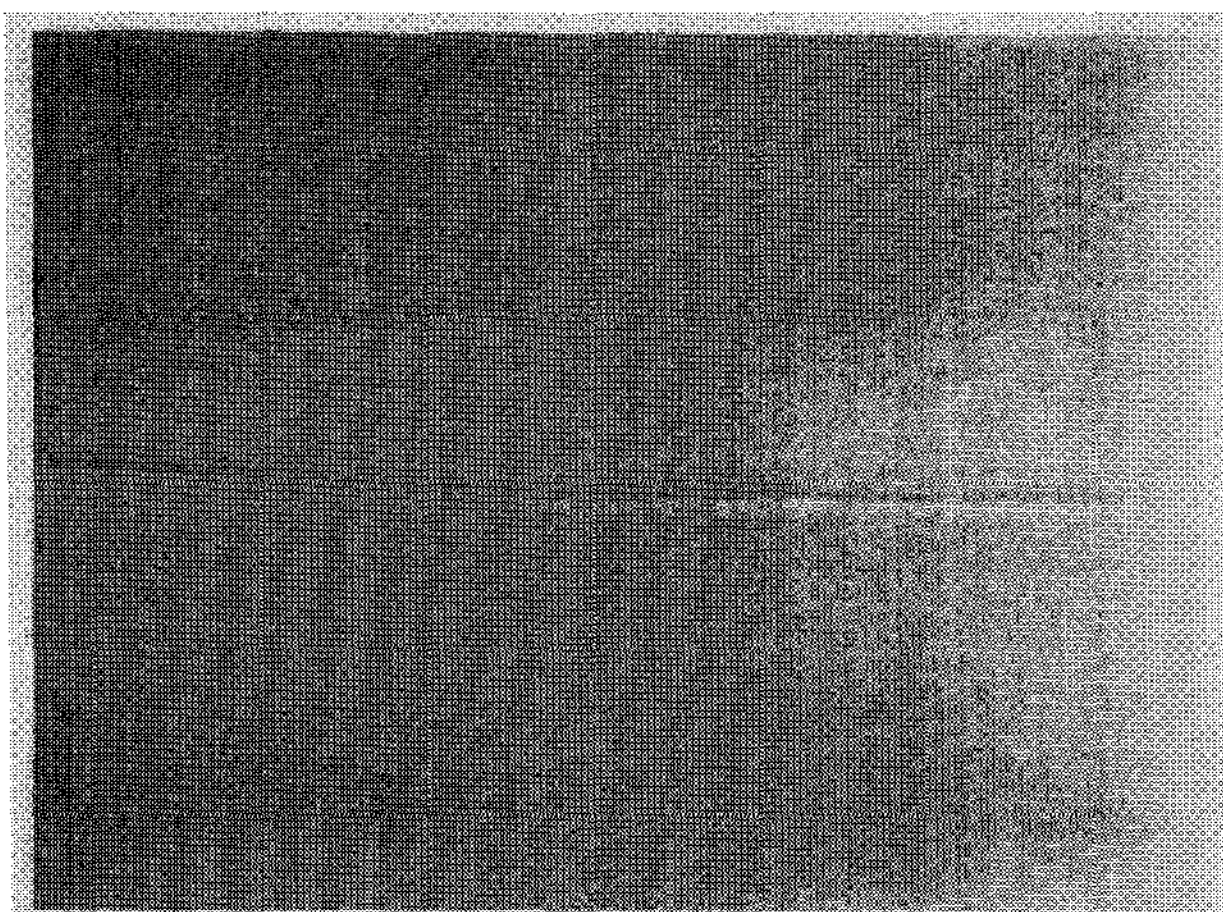


Fig. 9. Images of the polymer substrates covered ITO films (a) with a buffer layer and a novel-sputtering device and (b) without a buffer layer a novel-sputtering device.



(a)



(b)

Fig. 10. Images of the cracking and defects in the ITO films deposited on the polymer substrate (a) without a buffer layer and (b) with a buffer layer.

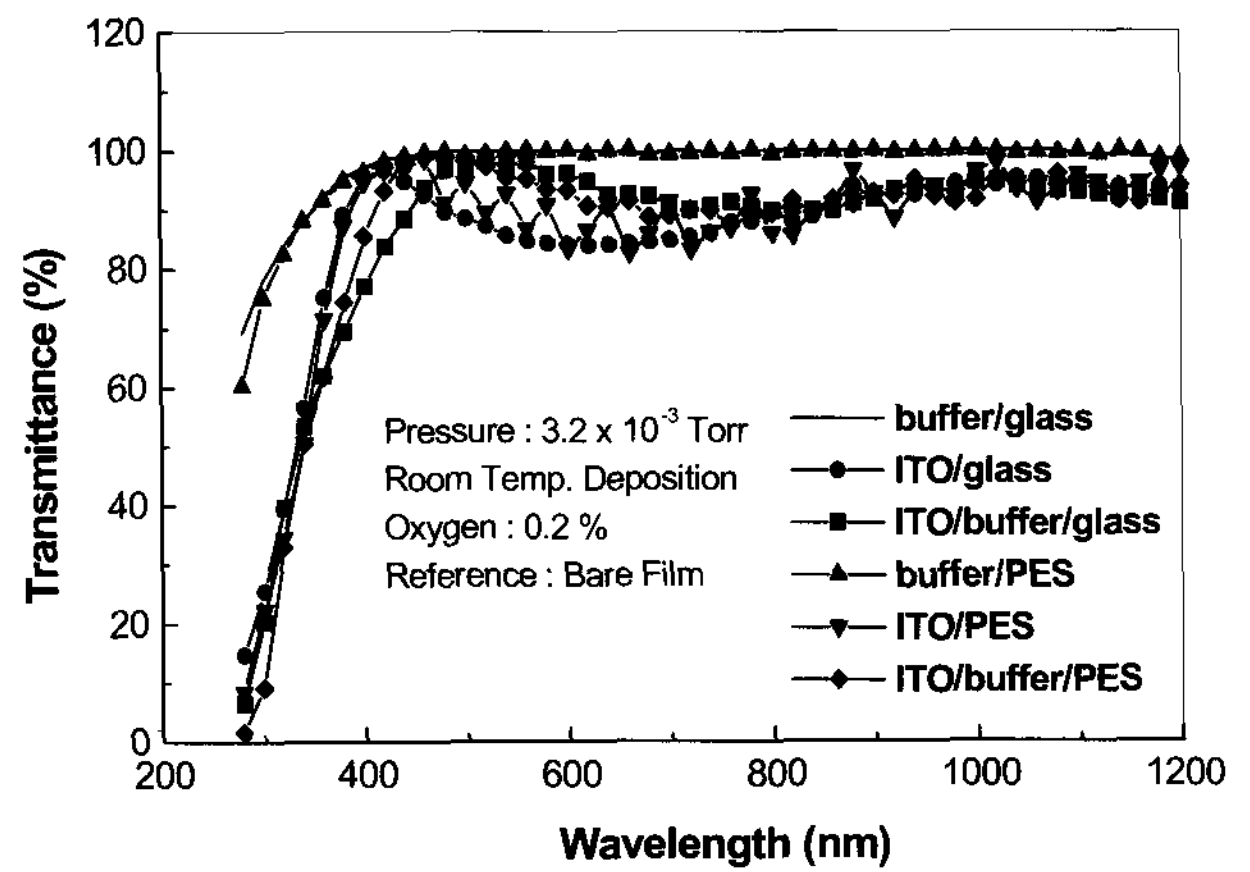


Fig. 11. Influences of a buffer layer on the optical transmittance in the ITO films.

TABLE 4. Specifications of a prototype color STN plastic film LCD.

Number of pixel	288 × 64
Number of color	256
Contrast ratio	8 : 1
Duty ratio	96
Pixel size	70 × 210 μm

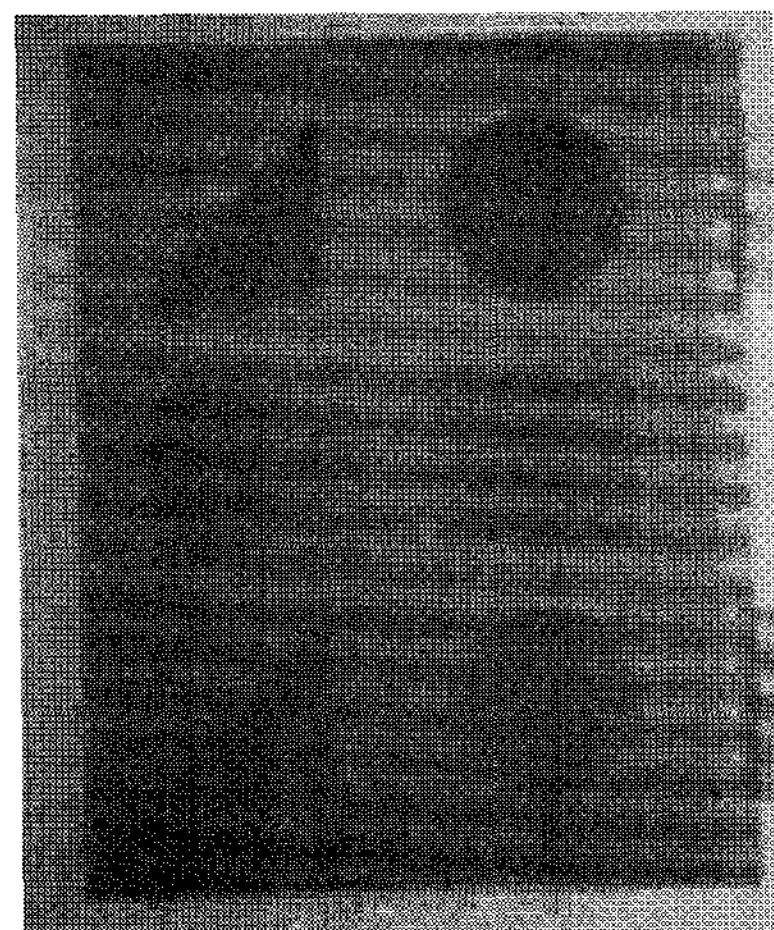


Fig. 12. Image of a reliable operation in the prototype color plastic film STN LCD.

3.5 Application to the color plastic film LCD

A prototype color plastic film LCD panel was fabricated using ITO thin film deposited on PES polymer substrate to confirm whether the panel could be realized in accordance with our experimental results. The specifications for the LCD panel are indicated in Table 4. The pixel pitch and size of the panel were 90 μm and 70

× 210 μm, respectively. The deposition conditions were the same to those of the experiments, and as shown in Fig.12, the test pattern was displayed well over the entire area of the display screen, showing that the ITO films deposited in this study was adequate for the application.

4. Conclusions

ITO films were rf-sputtered under various process parameters such as an oxygen partial pressure, annealing temperature and atmosphere, and then the optical, electrical and chemical properties of the films were examined. With regard to the oxygen partial pressure, there is an optimal pressure for both the high transmittance and conductivity. Though, there are some differences in the optimum oxygen partial pressure between the polymer substrates and the glass substrates. It is suggested that the difference is induced by the gas and moisture absorbed by the polymer material. The oxygen in the polymer matrix may flow out to increase the oxygen partial pressure during the deposition process. Besides this property, other properties of the polymer substrates include high thermal expansion and low thermal resistance. These distinct properties induced relatively high electrical resistance compared with that of the ITO films deposited on the glass substrates accompanying multiple cracking and poor adhesion during the deposition process. In this paper, we introduce a novel device and a process for diminishing the thermal stress of the ITO films on polymer substrates. This instrument eliminated a tensile force perpendicular to the direction of the stretching force in the polymer substrate. In addition, buffer layer not only flattened the surface of the substrate but also improved optical transmittance and mechanical property. Therefore, we think that this report on the highlighting subject, i.e., the ITO films deposited on polymer substrates will contribute to the improvement for the emerging plastic-based flat panel display, transistor and sensor technology.

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