High Conducting Large Area ITO Electrodes for Displays Prepared by DC Magnetron Sputtering

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High conducting ITO films were deposited by DC magnetron sputtering using ITO targets. At substrate temperatures of 200\(^\circ\) C and 300\(^\circ\) C ITO films were reproducibly prepared with resistivities of 1.9 x 10\(^{-4}\) \(\Omega\)cm respectively 1.4 x 10\(^{-4}\) \(\Omega\)cm at a deposition rate of 20 \(\AA\)/s. ITO films prepared at room temperature show after annealing at 200\(^\circ\) C in air, nitrogen or vacuum the same low resistivity of 1.9 x 10\(^{-4}\) \(\Omega\)cm when using optimum sputtering conditions. The influence of the deposition temperature on the electrical, optical and etching properties was studied and related to the structure of the ITO films. Details on an optimized in line sputtering system for the economic large scale production of ITO films are presented.

1. INTRODUCTION

High transparent and electrical conducting layers especially Indium-Tin-Oxide (ITO) are mainly used for photosensitive devices, solar cells, displays and different optoelectronic devices\(^1,2\). High throughput deposition systems and deposition processes are necessary to produce reproducibly among others ITO-layers on large areas with superior optical (T > 87 %, ITO on glass) and electrical (<200 \(\mu\)\(\Omega\)cm) properties without disturbing particle generation. With a DC-magnetron-sputtering process together with a well designed deposition system these requirements can be realized as will be shown in this paper.

2. EXPERIMENTAL DETAILS

The experiments were carried out in a Z 600 in line sputtering system using magnetron cathodes of the type PK 500 with a target size of 488 x 88 mm\(^2\). The composition of the hot pressed ITO target was 90 wt\% In\(_2\)O\(_3\) and 10 wt\% SnO\(_2\). The following main process parameters were used:

- Soda lime glass: 1 mm
- Deposition temperature: R.T.-300\(^\circ\)C
- O\(_2\)-partial pressure: 0-6 \(\times\) 10\(^{-3}\) mbar
- Sputtering pressure: 5,5 \(\times\) 10\(^{-3}\) mbar
- Sputtering power: 600 Watt, DC
- Deposition rate: 2 nm/sec

3. RESULTS AND DISCUSSION

Influence of O\(_2\)-partial pressure

In Fig.1 the specific resistivity and the transmission of 100 nm thick ITO films are shown versus the O\(_2\)-partial pressure during deposition. The influence of the O\(_2\)-partial pressure on the specific resistivity can be explained by two competing effects. With increasing O\(_2\)-partial pressure the crystalline phase of ITO films is growing\(^3\) leading to a higher mobility of the carrier as X-ray analysis shows. This is also known from Hall measurements\(^3\). On the other hand
Fig. 1: Specific resistivity and transmission of 100 nm ITO films versus O₂-partial pressure

Hall measurements show that with increasing O₂-partial pressure the carrier density is decreasing. The change of film properties caused by a one hour annealing in air at 200°C is correlated to a considerable increase of the crystalline phase especially of the In₂O₃(222)-orientation. On the other hand there is no change of the topography with increasing crystalline phase indicating a fine crystalline structure, compared with the ITO film prepared at a substrate temperature of 200°C (Fig. 2). Fig. 3 shows the spectral transmission of an annealed 100 nm thick ITO film on glass for the lowest sheet resistance of 19 Ω/□. The value of the mean transmission between 400 and 860 nm is about 87%. The spectral transmission of the same sample before annealing shows a 10 - 20% lower transmission value.

Influence of substrate temperature

Fig. 4 shows the specific resistivity and the transmission of an 100 nm thick ITO film on soda lime glass versus substrate temperature.

Fig. 2: Surface SEM photographs of ITO films

Fig. 3: Spectral transmission of 100 nm thick ITO films on glass
The ITO films were prepared at the same O₂-partial pressure of $2.5 \cdot 10^{-5}$ mbar where the films prepared at room temperature had the best film properties after one hour annealing in air at 200°C. The rapid decrease of the resistivity and the increase of the transmission above a substrate temperature of 84°C can be explained by the growing of the crystalline phases of the ITO films. This could be confirmed by X-ray diffraction measurements, as shown in Fig.5. As shown in Fig.2 the size of the grains grows also with increasing substrate temperature. At 300°C 1 μm long grains with a predominant (400)-orientation can be detected. Such large grains cause less scattering of the carriers because there are less grain boundaries.

Etching Behaviour

ITO-films for applications which need a patterning must have a good etching behaviour especially sharp etch steps without residues. For etching a standard iron containing hydrogen chloride acid was used. The investigations have shown that for deposition temperatures below 160°C the etch step is not sharp and shows underetching with a lot of residues due to the different etching behaviour of the amorphous and crystalline phases. Fig.6 shows a SEM photograph of an etched ITO film prepared at 250°C with a perfect etch step. At this temperature only the crystalline phase exists.

Fig.4: Specific resistivity and transmission of 100 nm thick ITO films versus substrate temperature

Fig.5: X-ray diffraction pattern of ITO films prepared at various substrate temperatures

Fig.6: Etch step of an ITO film prepared at 250°C

4. IN-LINE SPUTTERING SYSTEM

The experiences with the previously described sputtering process and the experiences with deposition processes and deposition systems for other large area applications, e.g.
depositions plants for the production of EL-Displays, have been applied by designing a new sputtering system for the production of high quality films. This sputtering machine meets the following requirements:
- uniform deposition over a large useable substrate area of 600 x 770 mm², for double side deposition
- high throughput of high quality ITO films; 22 m²/h for a 150nm thick ITO film
- constant deposition temperature up to 300°C
- low particle contamination of the substrates (soft pump down and venting specially designed transport system)
- continuous substrate flow
- fully automatic process control
Fig. 7 shows a cross section of this vertical, modular in line system with 3 pairs of opposite magnetron cathodes and a center heater in the process chamber for constant deposition temperatures.

5. CONCLUSIONS
The described DC Magnetron ITO sputtering process and the specially for ITO coating developed vertical in line system allow to produce high quality particle free ITO films with a throughput of 22 m²/hour. This high productivity and the achieved low electrical resistivities ( < 1.4 x 10⁻⁴ Ωcm), the high transmission in the optical region from 400-860 nm (T>87 %) and the good etching behaviour of the prepared ITO films meet the high requirements for the production of inexpensive large area LCD's with high resolution.

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7. REFERENCES