3523(57,(62),1'807,12;('326,7('86,15(&,9(&26(),(/0$(752163877(5,1*

D.R. Gibson, I.T. Brinkley, G.H. Hall, E.M. Waddell and J.M. Walls
Applied Multilayers L/&(&PPHUFH$QXH%DWWOH*URXQG$6$

Key Words: Magnetron sputtering; Optical coating; Surface metrology; Transparent conductive oxide

ABSTRACT

Magnetron Sputtering has many advantages over conventional evaporation processes for the deposition of transparent conducting oxides. The sputtering process is "cold", making it suitable for use on the widest range of substrates including polymers. Moreover, the drum format provides more efficient loading for high throughput production.

An additional advantage for deposition of transparent conductive oxides, such as indium tin oxide (ITO), is plasma driven oxidation of residual fractionated metal component thereby improving film conductivity. This also removes the need for post-annealing of films.

In contrast to previous reactive &sputtering strategies the Closed Field process does not require a separate ion or plasma source for activation. Neither does it require the vacuum chamber to be separated by vacuum pumps or baffles into deposition and reaction zones. Oxidation occurs on the target and all the way round the substrate carrier resulting in low absorption, no need for post annealing and reduced film electrical dependence on oxygen partial pressure.

The use of the Closed Field and unbalanced magnetrons creates a magnetic confinement that extends the electron mean free path leading to high ion current densities. The combination of high current densities with ion energies in the range ~30eV creates optimum thin film growth conditions. As a result the films are dense, spectrally stable, super-smooth and with excellent optical/ electrical characteristics.

This paper presents optical, electrical and surface metrology data for CFM sputtered ITO.

INTRODUCTION

ITO is used to make transparent conductive coatings. Thin film layers can be deposited by electron-beam evaporation or sputtering.

Typical applications of ITO-coated substrates include touch panel contacts, energy conserving architectural windows, defogging aircraft and automobile windows, heat-reflecting coatings to increase light bulb efficiency, gas sensors, antistatic window coatings, wear resistant layers on glass, cold mirrors, etc.

The deposition parameters play interdependent roles in the optimization of film properties. Principal among the deposition parameters are partial pressure of oxygen, substrate temperature, rate of deposition and material composition. Some processes require post deposition baking at 300-500° C in air to oxidize residual fractionated metal component and improve conductivity. For sputter processes, high-energy plasma can be substituted for a high substrate temperature[1].

Smooth transparent conducting oxides (TCO) thin films such as indium tin oxide are also important in LCD, OLED and electrochromic displays[2] and EMC coatings.

It has been reported previously [3,4] that "closed field" reactive magnetron sputtering produces dense, spectrally stable metal-oxide optical coating material with refractive indices typically close to that of the bulk material. In this paper we report new results that show CFM sputtering from a indium tin sputter target produces low stress, transparent conductive ITO thin films that are super-smooth with exceptional electrical and optical properties.

© 2006 Society of Vacuum Coaters 505/856-7188

REACTIVE CLOSED FIELD MAGNETRON (CFM) SPUTTERING

The reactive closed field magnetron sputtering process was developed in 1991 by Teer[5] and has been used successfully for some time in the field of tribological coatings[5]. Recently, the process control has been developed to enable sub-nanometre thickness precision[4,7] for application to multilayer optical coatings.

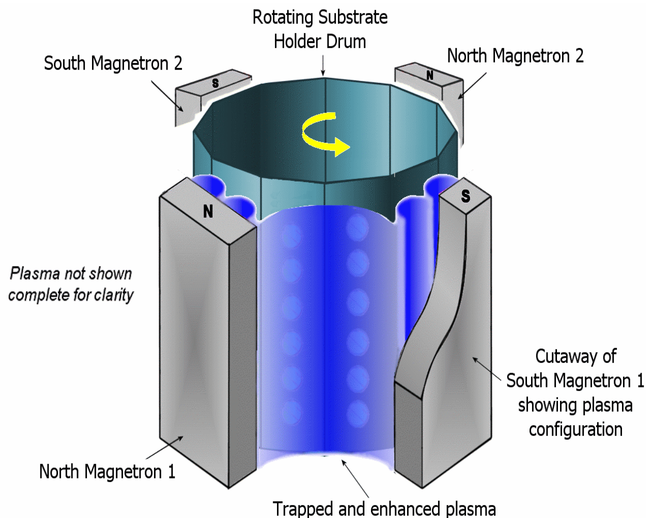


Figure 1. The "closed field" magnetron sputtering process does not need a separate ion source. Adjacent magnetrons are made opposite polarity to trap the plasma all the way round the drum. This ensures that all the metal deposited by the magnetrons is converted into metal-oxide.

The closed field process is illustrated schematically in Figure 1. The closed field configuration was developed to increase the ion current density in the plasma by arranging that neighboring magnetrons are of opposite magnetic polarity. Using this arrangement, the deposition volume in which the substrates are located is surrounded by linking magnetic field lines. This traps the plasma region, prevents losses of ionizing electrons, and results in significant plasma enhancement. The closed field system produces enhanced reactivity due to the high ion current density (>1mA.cm^{-2}). The ion energy is determined by the induced Voltage on the substrate carrier which is typically in the range (20-30)eV. This combination of high ion current density and low ion energy produce ideal conditions for growth of optical quality thin films.

During reactive oxidation in the closed field process, the oxygen is admitted into the unbalanced magnetron plasma. Target oxidation occurs but is controlled using target voltage control with feedback to the mass flow of oxygen. Surface charging is overcome by using pulsed DC power.

The key advantage of the process is that no separate ion source, plasma source or microwave ion source is required. Nor is it necessary to partition the working vacuum chamber into deposition and reaction zones. This simplifies the system, reduces cost, and improves reliability. It also makes it economical to scale the technology to virtually any batch size.

THE CLOSED FIELD MAGNETRON (CFM) SPUTTERING SYSTEM FORMAT

The thin film materials presented in this paper were deposited using a CFM450 system from Applied Multilayers LLC, the system shown in Figure 2. It uses a 250mm diameter vertical drum substrate carrier and up to four 405mm linear magnetrons. The drum is divided into a number of segments on which the substrates are mounted. Access is made through a large hinged external door.

The distance between the magnetron and the segmented drum surface is 100mm. Curved substrates are loaded behind apertures in the segment to ensure a reasonably constant average target to substrate distance.

The system used in these trials was pumped using a 2000 liters/sec BOC Edwards Diffstak. A Meissner trap is used for rapid removal of water vapor and reduced pump-down times.

Figure 2. CFM450 AR coating system from Applied Multilayers LLC.

The system is also fitted with a rotating shutter to expose the substrate to the appropriate target. The shutter is also used to enable targets to be conditioned without exposure to the substrates. A schematic of the drum/shutter configuration is shown in Figure 3.

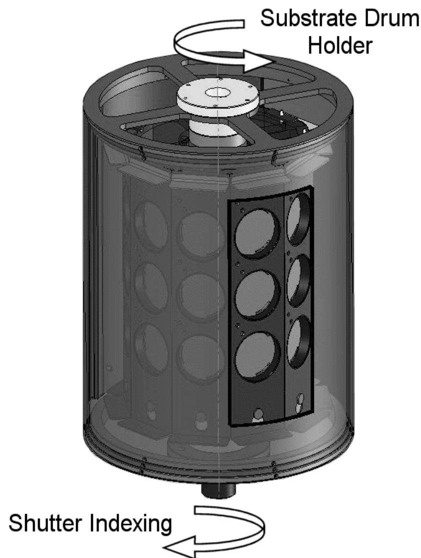


Figure 3. Drum/shutter configuration.

Deposition work below is based on use of a 10% tin (by weight) indium tin sputter target.

OPTICAL

The transmission of a 365nm thick layer of ITO is shown in Figure 4. CFM sputter deposition was from an indium tin target [10% tin content by weight].

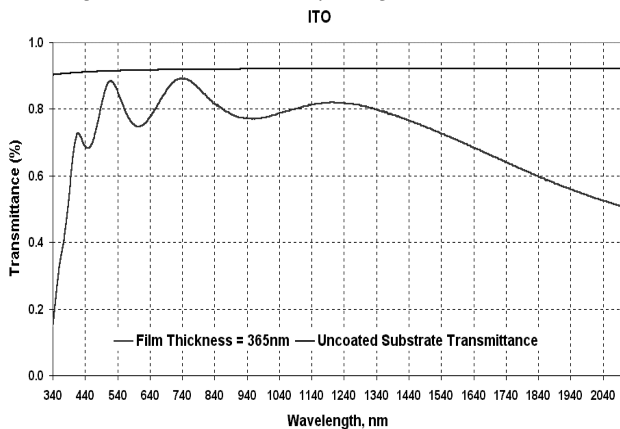


Figure 4. The spectral transmittance of a 365nm thin film of indium-tin oxide (ITO).

Figure 5 shows refractive index and absorption coefficient dispersive data for ITO.

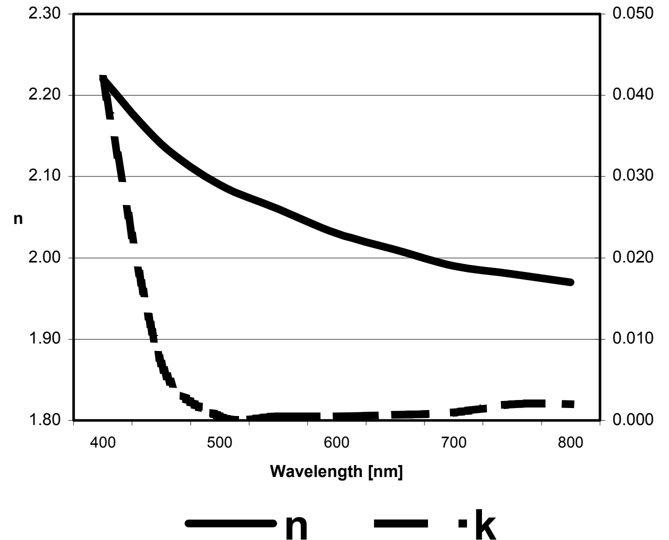


Figure 5. Refractive index and absorption coefficient dispersive characteristic for ITO – 10% tin content (by weight) sputter target

Wavelength minima in absorption coefficient can be varied by changing sputter target tin content.

Figure 6 shows data from room temperature deposited non-annealed indium tin oxide (ITO) deposited on to borosilicate glass. Use of a Nb_2O_5/ SiO_2 overcoat provides necessary index matching to maximise transmission over the visible spectrum. Average transmission > 97% is achieved over the visible region.

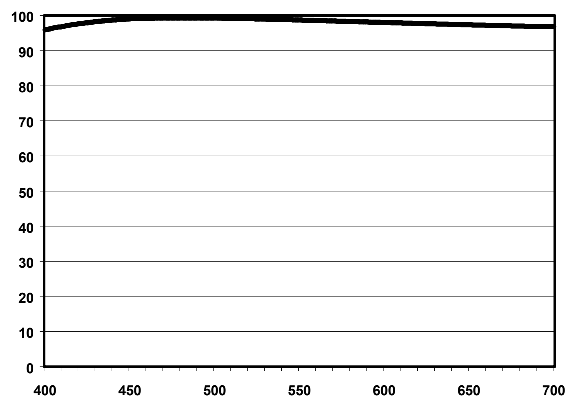
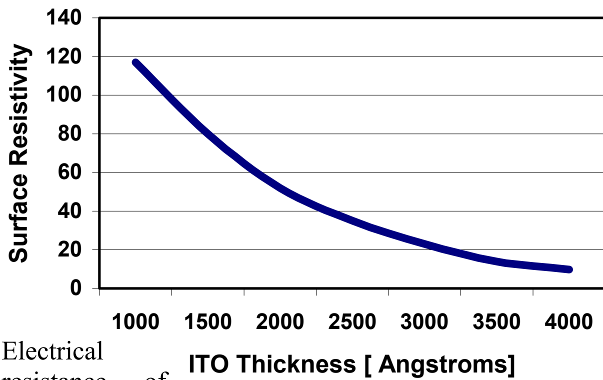


Figure 6. Spectral transmittance of Nb_2O_5/ SiO_2 overcoated ITO – index matched to reduce reflection loss.

ELECTRICAL



Electrical resistance of deposited ITO as a function of oxygen partial pressure is shown in figure 7.

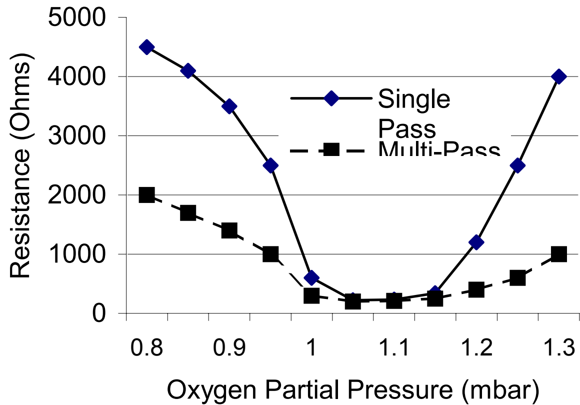


Figure 7. Electrical resistance of ITO as a function of oxygen partial pressure. Data shown for substrate single pass and multiple pass in front of magnetron.

Electrical conductivity is the product of carrier density and mobility. Conductivity for partial pressures < resistance minimum (conductivity maximum) is dominated by carrier density whilst for partial pressures > minimum resistance minimum is dominated by carrier mobility.

Optimum oxygen partial pressure corresponds to resistance minimum. Note for configuration with substrate undergoing multiple pass infrontof magnetron, as in equipment shown in figure 2, the resistance minima as a function of oxygen partial pressure is shallower. This is due to film oxidation occurring around the drum in addition to infrontof the sputter target[8].

Figure 8 illustrates surface resistivity (measured using a four point probe, and thickness evaluation from spectral transmittance analysis) as a function of ITO thickness. Deposition corresponds to use of oxygen partial pressure to minimize ITO film resistance.

Figure 8. Surface resistivity (ohm/sq) as a function of film thickness for CFM sputtered ITO – optimal oxygen partial pressure for minimum resistance utilized

SURFACE METROLOGY

Quantitative surface roughness assessment was carried out using Coherence Correlation Interferometry using a Talysurf CCI 3000A from Taylor Hobson.

The CCI technique is a non-contacting surface metrology tool. Its major advantage over techniques such as AFM is that it is fast and it takes its data from a relatively large, and hence more representative area, typically 300um x 300um.

The CCI combines a coherence correlation algorithm with a high resolution digital camera array to generate a three-dimensional representation of a structure by scanning the fringes through the surface in a “Z” direction and then processing the information to transform the data into a quantitative three-dimensional image with 0.01nm vertical resolution.

Figure 9 shows a CCI scan of a 365nm thick ITO film, deposited onto Schott D263 super smooth glass (rms roughness of uncoated glass is< 0.5nm).

© 2006 Society of Vacuum Coaters 505/856-7188
49th Annual Technical Conference Proceedings (2006) ISSN 0737-5921

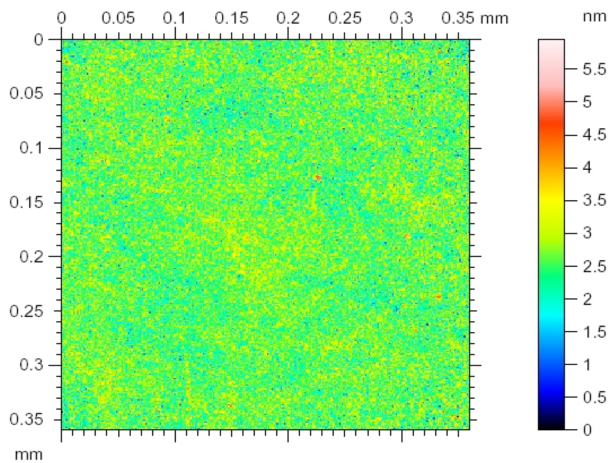


Figure 9. A CCI image from a 365nm ITO film with a measured rms roughness Sq=0.453nm.

The peak to valley roughness is 4.4nm and the rms roughness is 0.453nm. This is super-smooth and comparable with the original glass surface.

DISCUSSION

Closed field reactive magnetron (CFM) sputtering has been used for many years to produce highest quality tribological coatings. The same basic process produces transparent conductive ITO with outstanding optical and electrical properties. Also the process is capable of producing low stress, dense, super-smooth coatings with low optical scatter. These properties are all derived from the fundamental advantage of the closed field strategy inherent in the combination of high ion current density combined with low ion energy.

The CFM process can be exploited in batch format or in-line format. The process does not require an auxiliary ion or plasma source and without this overhead, the batch systems are scaleable to meet the demands for a small development system through to high throughput production systems. The in-line format is particularly appropriate to the high volume production demands for high quality ITO on LCD or OLED displays or EMC coatings on mobile telephones.

ACKNOWLEDGEMENTS

The authors are grateful to J Armstrong (Taylor Hobson) for the CCI 3000A measurements.

REFERENCES

1. A.G. Spencer, K. Oka and R.P Howson, *Vacuum,Vol 38, p 857-859,* 1988

2. D.R. Gibson, I Brinkley and J.M. Walls, *SPIE Proc 5618*, October 2004, p114

3. J.M. Walls, D.R. Gibson, I. Brinkley, D.G. Teer, P. Teer and J. Hampshire, *SVC 46th Annual Technical Conference Proceedings*, 2003, 387

4. D.R. Gibson, J.M. Walls, I. Brinkley, D.G. Teer, P. Teer and J. Hampshire, *SVC 47th Annual Technical Conference Proceedings*, 2004, 377

5. D.G. Teer, US Pat. #5,554,519, 1991

6. D G Teer Wear 251 (2001) 1068

7. D R Gibson and J M Walls Europhotonics 9 (2004), 32

8. A.G. Spencer, private communication.

© 2006 Society of Vacuum Coaters 505/856-7188