Automated reactive thermal evaporation system for transparent conductive coatings

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Abstract — This work presents fully automated plasma-enhanced reactive thermal evaporation system (rf-PERTE) that can be used for the deposition of transparent metal oxide films with high reproducibility of their electrical and optical properties. The developed hardware/software platform enables the full control over the critical deposition conditions such as mass flow of oxygen, process pressure, current flowing through crucible and rf-power. For indium oxide films on glass substrates a resistivity of $9 \times 10^{-4} \ \Omega \cdot \text{cm}$ and a transmittance of 90% in the visible spectral range were achieved without substrate heating. The system is also suitable for the deposition of transparent conducting coatings in a wide range of plastic substrates, for applications in the field of flexible sensors or solar cells. In particular, we have successfully deposited indium oxide on PEN (polyethylene naphthalate) sheets with electrical and optical properties approaching the ones of the films deposited on glass substrates.

Keywords: TCO, Deposition, Transparent Electronics.

I. INTRODUCTION

Transparent conducting oxide (TCO) layers on polymeric substrates are an important component of flexible electronics [1]. Substrate materials such as polyethylene terephthalate (PET) or polyethylene naphthalate (PEN) are being considered for opto-electronic devices due to their high transparency and low cost [2]. The technological challenge is that TCO coating should be deposited at low temperatures, desirably below the glass transition point for these plastics. Several vacuum techniques such as dc and rf sputtering, ion beam-assisted evaporation, and arc-discharge ion plating have been used for deposition of indium-tin oxide (ITO) on polymeric substrates [3-6]. However, the properties of low-temperature amorphous ITO on plastics are substantially inferior in comparison to crystalline ITO grown on glass substrates at high temperatures. To overcome the limitation on growing TCO films with satisfactory electrical and optical properties on plastic substrates, we have developed a radio-frequency plasma-enhanced reactive thermal evaporation (rf-PERTE) technique [7]. This work reports on an automated rf-PERTE system, which is suitable for deposition of In$_2$O$_3$ based coatings on unheated polymeric substrates.

II. DEPOSITION SYSTEM

Fig. 1 shows a general design of the rf-PERTE system. The system is based on a bell jar type vacuum chamber with a diffusion and mechanical pump vacuum pumping group. The typical configuration for thermal evaporation is used, with a distance between the tungsten boat and the substrate holder of 32 cm. For plasma assisting, an rf-electrode in the form of a copper ring is placed in the half-way between crucible and substrate holder. An electrically-driven shutter placed about 15 mm below the substrate holder, shields the substrate from oxygen plasma and impurities from the starting of the evaporation process. The oxygen injection into chamber is controlled by a SmartTrak 100 Series mass flow controller. A GenesysTM series programmable regulated power supply and a CesarTM Generator, Model 136, are used as dc and rf power sources, respectively.

A. Controller Hardware

All electronic units and electrical parts are linked to a microprocessor based control unit either through analog, digital or communication (RS-232) ports. The control unit is connected to a personal computer through USB interface that enables the system control using dedicated software. In order to keep the controller board as simple as possible, the analog to digital converter (ADC) included in the microcontroller was used. As seen in Fig.1, an analog input is used for the
measurement of the pressure signal coming from a Pfeiffer PKR251, the 10 bit resolution of the existing ADC was not sufficient for the determination of the process pressure with enough precision in the range of interest. To overcome this issue the pressure signal is converted in two steps with the aid of a differential amplifier and an analog output generated from a PWM signal from the microcontroller. In the first step the voltage from the gauge is measured on one ADC input, this value minus a fraction is then subtracted from the pressure signal and amplified 64x, the output voltage of the amplifier is then captured on the second ADC input and the pressure value calculated from the two ADC values. With this simple method the resolution achieved enables the accurate control of the pressure during the process.

B. Software

The control software was developed targeting the following tasks: the programmed control of all electronic units; real time monitoring of all process variables and their recording for post-analysis and documentation; automated control of critical process parameters; and the use of recipe files for process reproducibility. The user interface is intuitive and self-explanative, with the functional blocks for each piece of equipment, in a way that a rapid look is sufficient to get information about system status. A pressure chart is shown and updated in real time allowing the user to follow the pressure variations. When the initial values of the process parameters are reached, the program enables deposition in full-auto mode by starting the timer. In particular, a dedicated subroutine opens the shutter, holds the process pressure at a given value and adjusts the evaporation rate by varying the current through the tungsten boat. When the timer is run out, the shutter will be closed and with some delay dc- and rf-values will be set to zero to end the process.

C. Results

To produce transparent and highly-conductive InOₓ films, an optimal balance between the evaporated metal mass and absorbed volume of oxygen should be reached. This balance can be evaluated by measuring the pressure at steady oxygen flow, pumping speed, and RF-power. Fig. 2 shows that the pressure in the chamber decreases, when the evaporation of indium starts, and then stabilizes when the evaporation rate becomes constant. The deviation from stoichiometry in InOₓ was found to be related to the difference between the initial pressure, Pᵢ, and deposition pressure, Pₐdep, which is determined by the evaporation rate. The evaporation rate depends on multiple factors such as the dc power applied to the crucible, process pressure, amount of metal still in the crucible, etc. To stabilize the evaporation rate, the dc current through the crucible is automatically adjusted to keep the deposition pressure constant. This approach enables the reproducible deposition of metal oxide films with required stoichiometry simply by setting the differential pressure ∆P value

![Pressure in the chamber during the deposition.](image)

III. CONCLUSIONS

A simple technique for preparing undoped, conductive and transparent thin films of indium oxide has been developed using the rf-PERTE method. The resistivity of 9×10⁻⁴ Ω·cm was achieved for coatings on PEN and glass substrates. InOₓ films on glass and PEN substrates show 90 and 85% peak values of transmittance in the visible spectral range, respectively. Process automation proved to allow stable deposition conditions and high reproducibility of the fabricated film characteristics.

REFERENCES