

# Exploration into Sputtered ITO Film Properties as a Function of Magnetic Field Strength

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## ABSTRACT

Depositing high performing ITO films onto room temperature substrates has been an industry-wide challenge for decades. Identifying the process properties that drive film quality is the first key to developing advanced hardware capable of improving the ITO film properties. The room temperature ITO deposition process space and resulting film properties as a function of magnetic field strength are explored in this study to help determine the key mechanisms responsible for changing the ITO film properties.

## INTRODUCTION

Indium Tin Oxide (ITO) films have long been the cornerstone transparent conductive oxide for a variety of large area substrate applications. The deposition characteristics of the sputtering process and film properties have been extensively studied using a variety of techniques [1] [2]. Much of the prior research focused on changing the oxygen partial pressure, overall process pressure, sputter power level, and the target to substrate distance (TTS) [3]. These parameters are all important to the resulting film properties, but the optimization trends of these variables suggest that lower sputter discharge voltage processes are required for the optimization of the film properties. This trend is even more significant when the films deposited onto temperature sensitive substrates.

There are several process parameters that can be adjusted to reduce the discharge voltage: reducing the total sputtering power density, increasing the process pressure, overlaying a higher frequency (RF) power delivery signal, and modifying the magnetic field strength on the surface of the target. This study is focusing on the effects of the latter parameter.

Negative oxygen ions are often attributed to increasing the bulk resistance of sputtered ITO films as the energy of the ions is going to be proportional to the voltage of the cathode. Detection of negative

ions with a mass spectrometer during a large area production process does not appear to be a practical solution; thus an in-depth understanding the influences of process parameters using industrial acceptable techniques such as plasma emission monitoring can allow in situ process tuning. The effects of changing the magnetic field on the ITO film properties are explored in light of a broad spectrum plasma emission response.

## TESTING PROCEDURE

The primary goal of the experiments is to determine the effects of the magnetic strength on the ITO deposition process. To accomplish this goal, the experiments are designed to deposit ITO coatings while varying the oxygen gas flow for 3 different high strength magnet bars based on the Sputtering Components QRM magnet bar design, shown in Figure 1. The process pressure, target to substrate distance (TTS), deposition power, and substrate velocity are all kept constant.

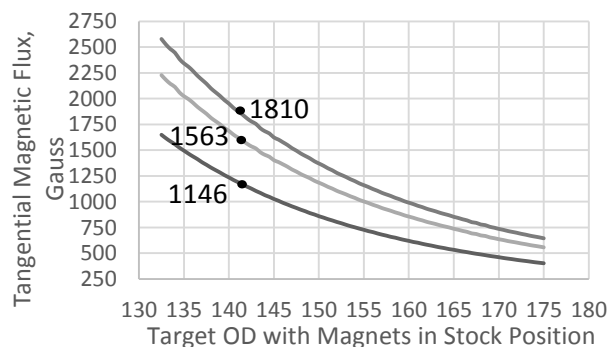


Figure 1: Tangential magnetic field strength for the 3 magnet bars used in the experiments

The experimentation test chamber is equipped with a hollow vertical drum that contains a rotating sample holder capable of indexing between 9 different samples. The drum is not actively cooled or heated resulting in the substrate temperature being a function of the heat transfer to the substrates and holders from the sputtering process. Each of the samples are passed by the cathode at 1 m/min for a single pass. The ITO films are sputtered

from a 650 mm long by 142 mm OD Umicore 90:10 ceramic indium tin target using a Sputtering Components TC-Series end block. An Advanced Energy Pinnacle 20 kW DC power supply is used to supply 6 kW to all tests. A 1000 sccm Alicat Basis mass flow controller is used to control the argon flow and keep the process pressure set to 1mTorr while a 100 sccm Alicat Basis mass flow controller is used to set the oxygen flow percentage for each experiment. The oxygen flow percentage settings are calculated using the oxygen flow divided by the total flow of argon plus the oxygen flow.

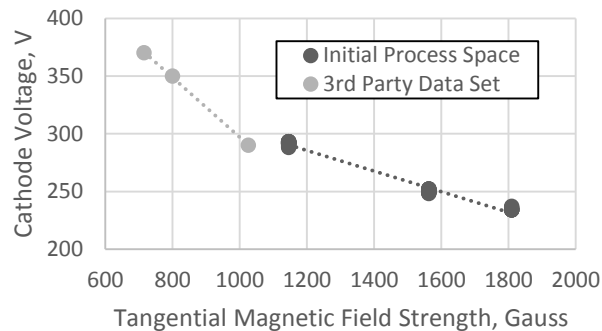
The oxygen flow percentages are varied from 0-4% in half percent increments with a full set of replicates. Prior to all experimentation the leak-up rates in the chamber are measured. Experimentation doesn't start until the leak-up rate is producing a calculated gas flow that is below 5% of the lowest oxygen flow setting used in the test plan. The leak-up rates are measured after running a 30-minute target burn in procedure to allow the chamber to be at the experimentation thermal condition which should negate any increased leak-up rates produce by the added thermal energy.

During the deposition process the plasma emission spectra data is monitored and recorded using a Plasus EMICON 1-MC plasma emission monitor. The optical data is collected from a single fiber optic with the end of the fiber positioned at the end of the cathode with a line of sight view through one of straightaways in the race track in the direction parallel to the target axis of rotation.

The coated borosilicate substrates are characterized post deposition using a Filmetrics F2-RT-UV Spectral Reflectometer to measure thickness, reflectance, and transmission. An Ecopia HMS-5000 + AMP55T is used to measure the sheet resistance, hall mobility, and carrier concentration. A Guardian Model SRM-232-100 surface resistivity meter is also used to measure the sheet resistance.

## RESULTS

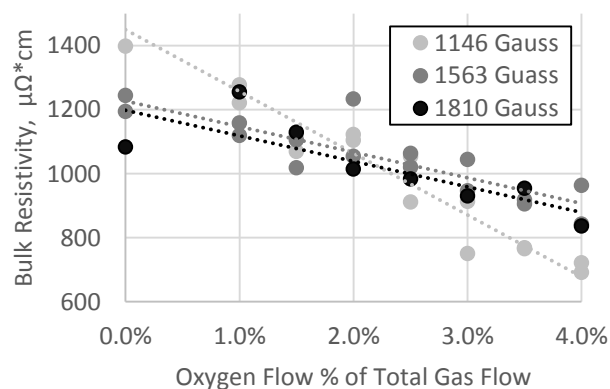
The voltage response for the 3 different magnet bars are compared to data collected by a 3<sup>rd</sup> party and plotted as a function of the tangential magnetic field strength in Figure 2.



**Figure 2: Cathode voltage as a function of the tangential magnetic field strength**

The 3<sup>rd</sup> party voltage data was collected at a process pressure of 2 mTorr instead of the 1 mTorr process pressure used for the initial process space data. The change in process pressure is responsible for reducing the voltage of the entire 3<sup>rd</sup> party data set in comparison to the initial process space.

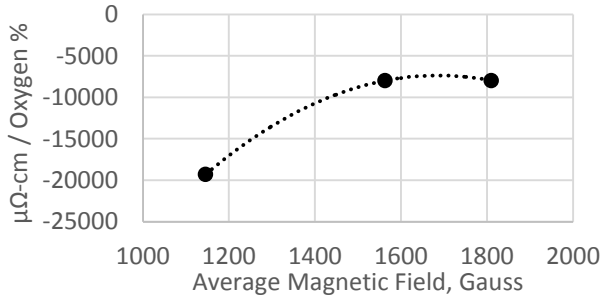
The narrow process conditions produced films with bulk resistivity values that ranged from 700 to 1400  $\mu\Omega\text{-cm}$  which are sufficient for the purpose of this experiment. The results of the bulk resistivity measurements are shown in Figure 3.



**Figure 3: Bulk resistivity as a function of oxygen flow**

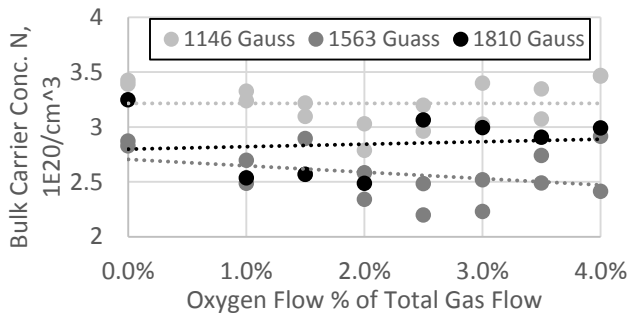
The bulk resistivity responses within the 0-4% oxygen percentage range all have negative slopes. The negative slopes suggest that the ITO has not been fully reacted with the oxygen and is closer to the target stoichiometry than to fully stoichiometric ITO. The slope of the bulk resistivity as a function of the oxygen flow percentage should be 0 at the local minima bulk resistivity, resistivity well, and when the ITO reaches full stoichiometry. Figure 4 shows that within the tested oxygen flow percentage range the bulk resistivity sensitivity to the oxygen flow as a function of the magnetic field strength is

reduced with increasing magnetic field strength. The bulk resistivity sensitivity to oxygen flow percentage is very similar for the mid strength and high strength magnet bar suggesting a possible response saturation or another dominant factor.



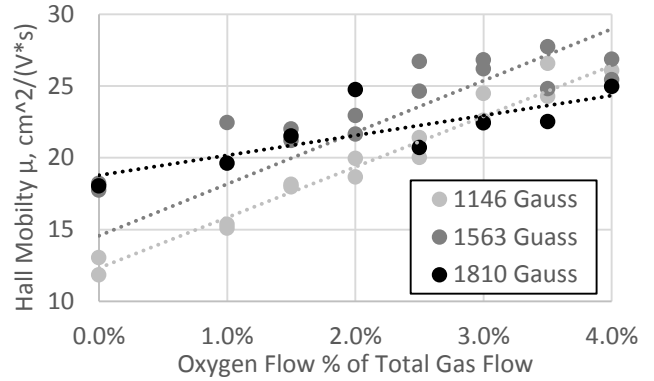
**Figure 4: Bulk resistivity sensitivity to oxygen % as a function of the average magnetic field strength**

Figure 5 shows that the bulk carrier concentration data has roughly flat slopes changing from positive to negative and back for the 3 magnet bar designs. A flat slope indicates that the bulk carrier concentration is not very sensitive to the oxygen flow percentage. The non-linear sensitivity indicates that there is an increased sensitivity to a factor other than the oxygen flow percentage or the magnetic field strength.

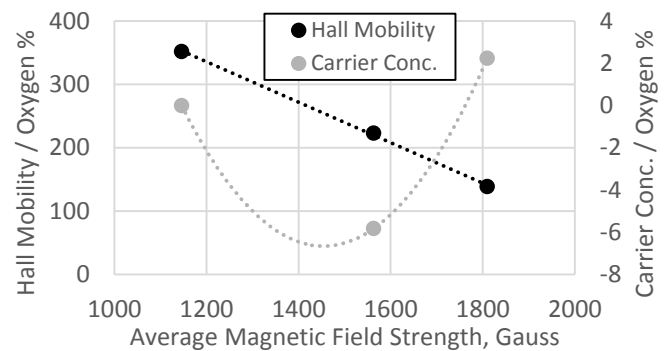


**Figure 5: Bulk charge carrier concentration as a function of the oxygen flow percentage**

The hall mobility response as a function of the oxygen flow percentage as shown in Figure 6, produced positive slope trends for each magnet bar strength. When the slopes of these responses are plotted as a function of the magnetic field strength in Figure 7, there is a linear relationship indicating that the magnetic field strength is a significant factor responsible for decreasing the sensitivity of the hall mobility to the oxygen flow percentage.

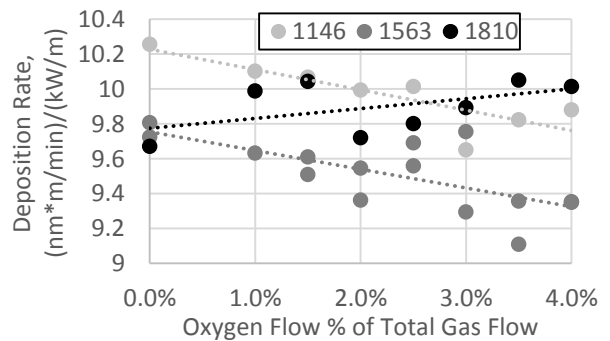


**Figure 6: Hall mobility as a function of the oxygen flow percentage**

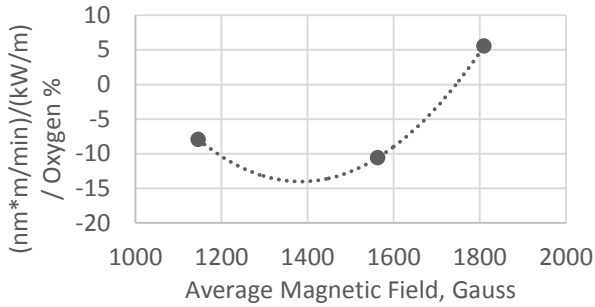


**Figure 7: Hall mobility and carrier concentration sensitivity to oxygen flow % as a function of the average magnetic field**

Figure 8 suggests that the power density normalized dynamic deposition rate decreases as a function of the oxygen flow percentage for the two lower strength magnet bars. The deposition rate increases with oxygen flow percentage for the strongest magnet bar. The dynamic deposition rate response sensitivity trend is shown in Figure 9. The non-linear sensitivity again indicates that there is another factor influencing the response sensitivity.

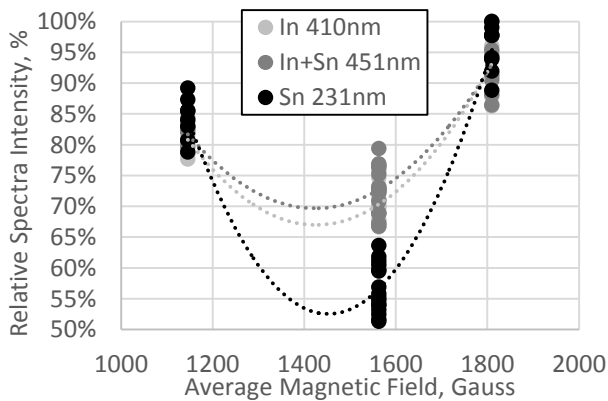


**Figure 8: Power density normalized dynamic deposition as a function of oxygen flow percentage**

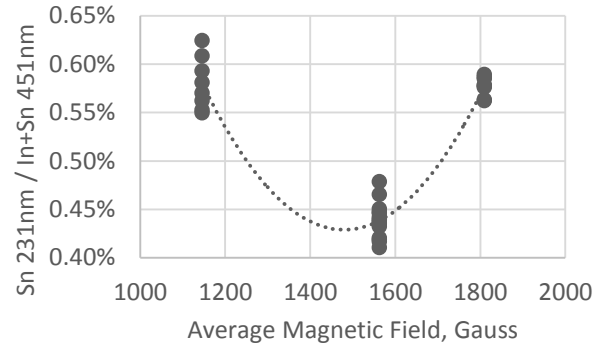


**Figure 9: Power density normalized dynamic deposition rate sensitivity to the oxygen flow percentage as a function of the average magnetic field strength**

The spectral responses from the plasma emission monitoring data starts to provide correlations between the previous response sensitivities. The indium 410 nm and Sn 231 nm spectral signals as shown in Figure 10 are similar to each other and have the same relative trend as a function of the magnetic field as the power density normalized dynamic deposition rate sensitivity in Figure 9. The In+Sn 451 nm response can be correlated to the deposition rate. The Sn 231 nm spectral signal in Figure 10 has a correlation to hall mobility sensitivity in Figure 7. This correlation suggests that the hall mobility sensitivity is related to the tin dynamic sputtering rate or the ratio between the Sn 231 nm and In+Sn 451 nm spectral emission counts as shown in Figure 11.



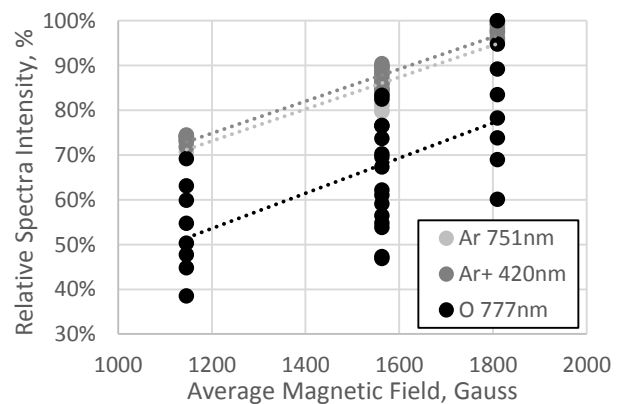
**Figure 10: Relative spectra intensity for the sputtered species**



**Figure 11: Spectral response for Sn 231 nm divided by In+Sn 451 nm as a function of the magnetic field strength**

The middle strength magnet bar responses in Figure 11 suggests that the tin 231 nm deposition rate is decreasing which is consistent with the deposition rate results. This decrease in the tin 231 nm signal could be related to the sputter yield properties of tin and the specific plasma impedance.

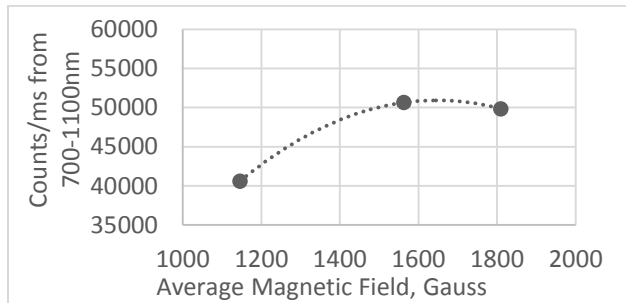
Figure 12 shows that the emission spectra response for the gas species increases linearly with magnetic field strength suggesting that the decreased plasma impedance due to the increasing magnetic field strength is increasing the amount of species that come in close contact with electrons in the plasma and release photons. The number of physical species is remaining constant so the plasma activation efficiency must be increasing linearly with the power supply current. This relationship can be further defined by the Corona Model.



**Figure 12: Gas species spectral intensity as a function of magnetic field strength**

The majority of the gas species visible in the plasma emission spectra are in the range of 700 nm to 1100 nm. Integrating the spectra over this range, Figure 13, provides a response that indicates the number

of activated gas species being produced by the plasma. The response in Figure 13 can be directly overlaid onto the response of Figure 4 suggesting that the integrated gas species spectral response can be used to calculate the bulk resistivity sensitivity to the oxygen flow percentage.



**Figure 13: Integrated spectral response from 700 nm to 1100nm as a function of the magnetic field strength**

The response in Figure 13 also suggests that more energy was delivered to the gas species with the mid strength magnet bar than with the highest strength magnet bar. When accounting for the conservation of energy the reduction in the sputtered species for the mid strength magnet bar must have been due to additional energy being put into the gas species produce in the spectra from 700 nm to 1100 nm, which is primarily composed of argon metastable species.

## CONCLUSION

The plasma emission spectral analysis of the ITO deposition process as a function of the magnetic field strength revealed that the plasma can shift the energy delivery to gas species other than the ions required for sputtering, this can dramatically alter the film properties. It also appears that correlations can be made between the spectra data and the deposition rate, carrier concentration sensitivity to oxygen flow percentage, and bulk resistivity sensitivity to the oxygen flow percentage. These correlations can be further used to determine the sensitivity of the process to other variables such as process pressure, target to substrate distance, target temperature, and deposition power. Lastly with more development these relationships may be able to be used to characterize film properties during the deposition process.

## REFERENCES

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- [2] M. Dudek, O. Zaveida, J. Klemberg-Sapieha and L. Martinu, "Ion-induced Effects During Reactive Sputtering of ITO Films," in *Society of Vacuum Coaters*, 2005.
- [3] M. Chuang, "ITO Films Prepared by Long-throw Magnetron Sputtering," *JMST*, vol. 26, pp. 577-583, 2010.