Chapter 1 Introduction

1-1 General Background

Display technologies have become an important Hi-Tech industry in recent years. In Taiwan, here comes a new policy called: ”Two Trillion and Twin Star Industries Development Plan”, which represents the importance and the attention for display field. Nowadays, the a-Si:H TFT, which is used widely as drivers of Active Matrix Liquid-Crystal Display (AMLCD), has encroached on the territory of the cathode ray tubes. But the deadly issue to the material based on a-Si:H in channel layer in TFTs is low field effect mobility (~0.5$cm^{2}\cdot V^{-1}\cdot S^{-1}$), photo sensitivity (low band gap about 1.7eV) and rather high deposition temperature (~400℃). Since the band gap of a-Si is in visible regime, the photo excited carriers (photo current effect) might make the array of AMLCD out of control. For this reason, the opaque metals to keep a-Si based channel blind from visible light are integrated necessarily. This causes lower opening of AMLCD pixels and more complicated device fabrication.

In terms of power consumption, a large part of the energy of the display is cost from the backlight instruments, such as Cold Cathode Fluorescent Lamps (CCFL). We need to maximize brightness and efficiency, but the opaque TFTs

based on a-Si:H restricts the amount of light that can be transmitted to the observers [1]. Then, fabricating high-performance devices is challenging of owing to a trade-off between the brightness and power consumption. For the purpose of enhancing power efficiency, display technology based on organic light emitting diodes (OLED), including polymers light emitting diode (PLED) are demonstrated for promising for providing lightweight, power efficient, and high brightness performance at reasonable voltage and current levels.

Another advantage of using organic and polymer material is the low fabrication temperature, which can also realize the array on flexible substrate (processing temperature below 200℃. But the challenging facing the OLED and PLED is the need of high driving voltage and high driving current for the controlling circuit. However, the driving circuit seems difficult for us to use the a-Si based material for the low mobility limited.

1-2 Motivation

One method to achieve the purpose of efficient power consumption, low fabrication temperature, and the recent progress in transparent oxides which are semiconducting to near-metallic materials is to develop Transparent Conducting

Oxide. TCO has been studied for several years, including Indium Tin Oxide (ITO), Tin Oxide (SnO_2), Zinc Oxide (ZnO), and etc.

We study and develop TFTs by substituting traditional amorphous silicon based from a popular TCO material—ZnO as channel layer. ZnO film deposited at room temperature reveals polycrystalline with a hexagonal wurtize structure and has a preferred orientation with the c-axis perpendicular to the substrate. Un-doped ZnO film behaves as an n-type transparent semiconductor due to the defects such as zinc interstitials and oxygen vacancies. It has a wide band gap (~-3.37eV) with optical transmission about 75% in the visible portion of the electromagnetic spectrum [2]. The main advantage of using ZnO deals with the fact that it is possible to growth high quality polycrystalline ZnO films at room temperature [3]. Besides that, thin films based on ZnO have been studied for several years for their low cost, low photo sensitivity, no environmental concern, and especially high mobility [4]. TFTs using ZnO as channel layer usually exhibit normally-on state, because the excess carries are over 10^{17} cm^{-3} in as deposited states [5].

Recently, there are a large number of teams announced the reports on the fabrication method on ZnO based TTFT by using RF Magnetron Sputtering [6], chemical solution deposition [7], pulsed laser deposition (LPS) [8, 9], and Atomic Layer Deposition (ALD) [10] instruments. However the electrical characteristics of

those reports were not good enough for producing commercial products nowadays. We need a better method and condition to improve the characteristics of ZnO films to achieve a higher performance TTFTs.

Zinc Oxide, having a hexagonal wurtize structure, has a variety of optical and electrical properties depending on deposition condition [11]. In general, the structure, electrical properties, Zn1-xO composition, and growth rate of reactively sputtered films are strongly affected by various processing conditions such as the geometry of the apparatus, plasma conditions, gas phase composition, processing temperature and annealing temperature.

In this paper, we studied the device properties of TTFTs with ZnO film as channel layer deposited by using DC plasma of an argon and oxygen mixture atmosphere with a Zinc disc target. Studies will be undertaken to interpret the growth mechanism of ZnO films, crystallographic structure, and electrical properties of the films. We have explored experimentally as functions of the deposition and annealing conditions and defined an optimal deposition condition for TTFT. By considering the carrier concentration, the mechanisms of film growth and treatment methods will be reported and discussed later.

Chapter 2 Experimental Procedure

Table 2 shows experimental flow path in my experiment. There were seldom papers about this deposition method for depositing ZnO film for TFTs utility. Because of the innovation of DC sputter, we got to reference lots of surveys on other deposition and treatment methods. Finally we defined a suitable deposition condition.

2-1 Transparent Conducting Oxide

Due to the characteristics of wide range of applications both in industry and in research, there came a large number of studies on transparent and highly conducting semiconducting oxide (TCO). The first TCO film was reported by Badeker [12], who used the film of Cadmium Oxide (CdO) as the main material. This film was prepared by thermal oxidation of sputtered films of Cadmium. CdO films were reported to be transparent, as well as conductive. There are still some reports about these kinds of transparent and conductive thin films, such as the oxidation material of Au, Ag, Cu, Fe, etc. But for the reason of bad stability and the characteristics changing with time, these thin films could not be used widely.

However, there are many extensive research work done to establish their utilities for device applications. The reports of the growth and characterization of semiconducting transparent conducting oxide films have been reviewed by many workers at various times. Holland [13] reported semiconducting material which was carried out up to 1955. Vossen [14], and Jarzebski and Marton [15] gave comprehensive reviews of experimental work reported up to the mid-1970s. Manifacier [16] et al and Dawar and Joshi [17] reviewed detail surveys of the work in this field up to the early 1980s. More recently, Hamberg and Granqvist [18] reported the work on Indium Tin Oxide films in detail, particularly from an application point of view [19].

There are some properties of Transparent Conducting Oxides at room temperature in ***Table 1*** [19]

2-1-1 Indium Tin Oxide

Tin-doped Indium Oxide (ITO) films which can be deposited by various techniques are always polycrystalline and retain a crystal structure of bulk-undoped In_2O_3. In general, the ITO films exhibit strong (1 1 1) or (1 0 0) preferred orientations depending on the deposition condition. There are many

reports about that the properties of the ITO films are strongly depends on the deposition condition and treatment after deposition, such as the partial oxygen pressure in the sputtering process when depositing ITO films [19], or annealing. Assumed that the band structure of undoped and Tin-doped In_2O_3 were showed in ***Figure 2-1***. Shaded areas denote occupied states. Shift of the bands is apparent. Band-gaps, Fermi wave number (kF) and dispersion relations are also indicated [18].

For achieving the goal of fabricating complete transparent thin film transistors, we try to replace metal 1 and metal 2 layers from TCO based materials. For the transparent and highly conducting requirement, we choose ITO as the replacement. Because of the reports on the influence of oxygen pressure in sputtering, we change the pressure of oxygen pressure during depositing our ITO films. We also anneal these ITO films after deposition. Then, we find an optimal recipe of the flux of oxygen in 0.3sccm and the annealing up to 400℃ to get the most conductive and uniform film.

2-2 TFT Fabrication

In our present work, the fabrication and properties of bottom-gate-type

thin-film transistors using an n-type ZnO film as channel layers will be described. Our ZnO-based TFTs, as shown schematically illustrated cross-sectional and planed views in ***Fig. 2(a)*** and ***2(b)***, were fabricated on SiO_2/n-Si substrates. Thermal oxide was chosen as the gate insulator. The channel layer based on ZnO was realized by using standard wet photolithography processing. The width and length range from 200 um to 1000 um, and the width/Length ratio of our TFTs was about 1 to 5.

Prior to the deposition of ZnO films, 300-nm-thick SiO_2 layers were thermally grown by 650℃ in Horizontal Furnace on n-type Si (100) substrates. This process was done in Class 10 in National Nano Device Laboratories. The 160-nm-thick un-doped ZnO films were subsequently deposited by sputtering pure metal Zn target (99.995%) in a dc power sputtering system at room temperature. The film thickness was measured by N&K analyzer 1280. The ZnO films were patterned via wet photolithography. Then, a shadow mask was subsequently used to pattern Al source/drain electrode pads deposited upside of the ZnO channel by thermal evaporation system, and the thickness of the electrons were about 300 nm.

2-3 Experimental Procedures

Thin films deposited procedures have been studied for a long time. We can divide the thin film deposited procedures roughly in two parts. One is Physical Vapor Deposition (PVD), and another one is Chemical Vapor Deposition (CVD) Technique. Since 1963, many works with the aspects of sputter deposition of metal films in various systems and configurations [20]. In this study, we use a Physical Vapor Deposition system－DC Sputter to deposit the ZnO films as channel layer and the TIO films as metal layer. A schematic diagram of the system is shown in ***Figure 3***.

The work function of DC sputter is described about that the direct current (DC) power is used for the sputtering deposition of conducting films, and for the substrate biasing of conducting films. There are both conventional DC power and pulsed DC power for the sputter deposition of coatings. DC sputtering is for applications starting with the deposition of numerous metallic films and with the reactive sputter deposition of conducting hard coatings such as the nitrides of Titanium, Zirconium, Hafnium. Most recently, there are new techniques with development of high power pulsed magnetron sputtering (HPPMS) using very high power DC pulsed that result in a very high degree of ionization of the sputtered materials.

2-4 Methods of Depositing ZnO Films

2-4-1 Changing DC power in sputtering system

ZnO films were prepared by reactive pre-sputtering with a direct current power in mixed argon and oxygen gas at atmosphere at a constant pressure of 7.6×10^{-3} Torr. As the sputtering apparatus a diode-type reactor equipped with a substrate holder, DC power supply, gas lines, vacuum gauges, and a pumping system was used. By using a machine pump, a residual pressure of the order of 2×10-6 Torr was achieved. A disc of zinc 4 inches diameter and 6 mm thickness with a purity of 99.995% was used as a target.

We change the power of pre-sputtering and sputtering to adjust the optimal uniformity of our zinc films. Pre-sputtering is used to clean the surface of zinc target. When pre-sputtering, we close the shutter to keep the surface of our sample from the particle generated by the procedure of pre-sputtering. The results are listed on the ***Table 3***. So we define the optimal power condition on pre-sputtering/sputtering as (45 / 60) W.

2-4-2 Adjusting the Rate of Argon and Oxygen

The experimental conditions are summarized in ***Table 4***. Among a number of deposition conditions which can obviously influence the properties of ZnO films, the concentration of oxygen was discussed in this paper. We can change the channel layer, including Zn/ZnO, $Zn_{1+x}O$, and ZnO, to achieve an optimal condition for TFTs We use N&K analyzer 1280 to measure the thickness of ZnO films. All those films were examined by X-Ray diffraction analysis (XRD) and Fourier Transform Infra-Red (FTIR). Besides the analysis of the ZnO films, we have also developed a Bottom-Gate type TFTs. The result of electrical properties will be shown on the next chapter.

2-4-3 Patterning

Many reports on the chemical durability of ZnO films are insufficient. Most of the ZnO based TFTs reported in the literature were fabricated by hard mask or lift-off processes [21-22]. These processes are difficult to achieve TFT-array, because the size is too big to fit the requirement of AMLCDs.

ZnO films were reported to be patterned by photolithography and wet-etching with HCl and HNO3 [23]. But the etching rate is too fast to directly use HCl or HNO_3 in thin films wet photolithography. There are so reports about

using CH_3COOH solution which can slow etching rate [24-25]. In our studies, we add CH_3COOH in HCL and HNO_3. After large number of testing, we find out the optimal solution rate as H_2O: HCl/HNO_3: CH_3COOH = 40: 1: 1.

2-4-4 Changing Annealing Temperature

In general, the physical characteristics of polycrystalline semiconductors are easily modified by their crystallite sizes and boundary effects. The electrical properties are much more easily disturbed as long as the band structure altered inside the crystallites from the optical properties. The size of crystallites and the magnitude of the associated grain-boundary effects are dependent upon the preparation and treatment method, such as temperature in deposition or annealing, sputtering process, and deposition technique. In this section, we adjusted the annealing temperature for ZnO films to find out the optimal conditions for TTFTs.

Annealing at Atmosphere in Room Pressure Furnace

Room Pressure Annealing Furnace was used as annealing equipment at beginning for the advantage of easy operation. Samples we chose were sputtered at room temperature (RT) on the silicon substrates which were

performed in a mixture of oxygen In process, nitrogen gas was flowing in the furnace with 166.67sccm to keep the environment in a clean nitrogen gas and protected samples from affected by other gas in environment. We put samples with sputtered in argon/oxygen rate as (24 / 5),(24 / 8), and (24 / 10) in 300℃ and 500℃ temperature environment for 1 hrs.

Annealing with Vacuum Annealing Furnace

Vacuum Annealing Furnace was used as annealing equipment. Samples could be studied with only temperature influence in high vacuum atmosphere, or in a pure oxygen or nitrogen atmosphere by using vacuum Annealing Furnace. We controlled the experimental parameter only about temperature to change the properties of ZnO films. We put the samples in sputtered with (argon / oxygen) rate as (24 / 3), (24 / 4), (24 / 5), and (24 / 6), four condition in 300℃, 400℃ and 500℃ temperature in high vacuum environment for 1 hour. Then we did some film measurement about the as-treat-samples and measured the electrical properties to define the semiconductor characteristic.

2-4-5 changing Annealing Pressure With Vacuum Annealing Furnace

Vacuum Annealing Furnace was used as post-treatment equipment. Thermal annealing after the as-deposition with (argon / oxygen) ratio (24 / 4), and (24 / 5) condition in the pressure of 0.05, 0.1, 0.15, and 0.2 mtorr with pure oxygen flow for a hour. Then we did some film measurement about the as-treat-samples and measured the electrical properties to define the semiconductor characteristic.

In this method, we hoped to utilize oxygen flow to cover the imperfect of the ZnO lattice to achieve the semi-conductive behavior for our TFTs.

Chapter 3 Results and Discussions

3-1 ITO Film Analysis

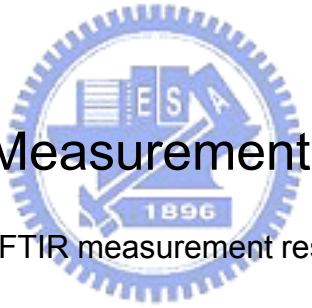
We can see the result of film sheet resistance in ***figure 3-1-1*** and ***figure 3-1-1*** by using four-point probes measurement in NDL. We found the deposition rate and conductivity of as-deposited films related to the power applied by the DC system. Low deposition rate / oxygen flow rate ratio causes low resistance films. Oxygen partial during the deposition strongly influence the electrical properties. Electrical properties related to oxygen vacancies which can increase the conductivity of ITO films. Oxygen vacancies give rise to a shallow donor level just below the conduction band, and increase the carrier concentration. [18.1]

3-2 ZnO Film Analysis

3-2-1 The XRD measurement of ZnO film

Figure 4 and ***Figure 5*** illustrates the measurement of as-deposited ZnO film on a single crystalline Silicon substrate (100) with different argon and oxygen mixture, and the samples of (argon / oxygen) ratio (24 / 4) annealed

in different temperature. Only (002) peak at 2θ=34.2 degree was shown on the ZnO film. The XRD measurement result can indicate that preferred orientation of the ZnO film is c-axis perpendicular to the substrate. This result also fits to the reports from other papers [26]. On the result of ***Figure 5***, we found that there are some shifts on peak of XRD with the increasing temperature. We determined the morphology as the tensile strain produced within the film caused by thermal effect.



3-2-2 The FTIR Measurement of ZnO Film

Figure 6 illustrates the FTIR measurement result of as-deposited ZnO film on a single crystalline Silicon substrate. The principle of FTIR is that the energy of the molecules of our samples in unoccupied excited rotational and vibration states, which is different from the ground-states, can correspond to photon energies found in the infrared. These can be detected and identified by FTIR [27]. There are two peaks in the wave number (cm^{-1}) of 407 and 576. We have compared these peaks to the ZnO film deposited by RF sputter by sputtering ZnO target, and they have the same result. Because the FTIR is not suitable to symmetry structure like ZnO, there are seldom reports about the

measurement of ZnO films by using FTIR. We can see the comparison in ***figure 6, 7***. The affects of changing (argon / oxygen) rate or the annealing temperature cannot be differed from each other. We determined the result because the measurement equipment (FTIR) can only used for asymmetric structure, but the structure of ZnO belong to symmetric structure. That is the reason we determine FTIR is un-suitable for us for measuring ZnO film.

3-3 The electrical Characteristic of ZnO Based TFTs

3-3-1 Annealing in room pressure states

Figure 8 shows the I_D-V_G our ZnO-based TFTs without annealing and in 300℃ 、500℃ with *Room Pressure Annealing Furnace* and has been normalized. ***Figure 9*** shows the I_D-V_D of our ZnO-based TFTs annealing in 300℃. The sample was fabricated in argon/oxygen rate as (24 / 5) sccm by sputtering. The other samples, such as rate of (24 / 8), (24 / 10) presented an insulator characteristic. The on/off ratio of the ZnO-based TFT is about 2×10^{-4}. The mobility is about 0.018 $cm^2.V^{-1}.s^{-1}$. Vth is about 22.2V. This result

is too far away from the commercial products based on amorphous silicon, so we want to change our condition by using Vacuum Annealing Furnace. But we can define a condition to deposit ZnO as semiconductor property.

3-3-2 Annealing in high vacuum states

Figure 10 shows the I_D-V_G of the ZnO-based TFTs without annealing and in 300℃、400℃、500℃ with Vacuum Annealing Furnace annealing. ***Figure 11*** shows I_D-V_D relationship in 300℃ annealing conditions. The sample was chosen (argon / oxygen) rate in (24 / 4) sccm.

In ***figure 10***, we found something interesting. When the annealing temperature below 300℃. Heat effect seems cause the broken of weak bonds, and makes traps generated. With the traps increasing, the on-current and off-current decreases at the same time. Then the mobility decreases.

When the annealing temperature above 300℃. Heat effect causes the desorption of Oxygen. With the decreasing of oxygen partial, the oxygen vacancies generated. For the respect to conductivity of ZnO properties, the increase of oxygen vacancies will increase the carrier concentration. Then the

ZnO films may serve as conductor behavior.

3-3-3 Annealing with oxygen flow

Figure 12 shows the I_D-V_G of the ZnO-based TFTs annealing in 0.05 mtorr, 0.1mtorr, 0.15 mtorr, and 0.2 mtorr with Vacuum Annealing Furnace. ***Figure 13*** shows the relationship between annealing pressure and mobility changing. We found the mobility decreased with the increase of the oxygen flow in furnace. Above the pressure of 0.15 mtorr, the mobility decreased dramatically. We figured out that the reason is the generation of oxygen interstitial in back channels of the ZnO films. Oxygen interstitial would cause the decrease of the conductivity of ZnO film.

In 0.15 mtorr, one hour annealing conditions, the concentration of oxygen seems too low to affect the channel layer, and only the back channel layer had been influenced. So, only the off-current had been decreased. With the oxygen concentration up to 0.2 mtorr, there was enough oxygen concentration which could cause the decrease of on-current. Then the mobility would decade at the same time. But on the other hand, with the decreasing of off current and the unchanged of on current in 0.15 mtorr,

annealing for one hour condition, the on-off ratio increased over one order. That was an un-anticipated advantage we got in this experiment.

Chapter 4 Conclusions

We have developed an optimized deposition condition for sputtering ZnO semiconductor film and succeeded to fabricate a ZnO-based transparent thin film transistor with bottom-gate structure. The optimal conditions for depositing the ZnO film by DC sputtering at room temperature is under the atmosphere of (argon / oxygen) mixture (24 / 4) sccm. With the development of wet etchants, ZnO active regions can be patterned exactly and completing TFT device fabrication.

Followed by the implementation of thermal annealing at different temperature, and in different pressure with oxygen flow, the mechanism and the effect of thermal treatment on ZnO films also were studied in detail. High temperature annealing process seems to cause the oxygen desorption effect, and increase the conductivity dramatically. In addition, high concentration of oxygen ambient would cause the decrease of on-current and off-current. The most suitable deposition condition for ZnO films with DC sputter system have been successfully established by annealing ZnO at 300℃ and with 30 sccm oxygen flow at 0.15 mtorr. In this work, the proposed technology with DC sputtering at room temperature is feasible and compatible with the production

line technology at present.

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Figure1-1 Motivated Transparent Thin Film Transistor device

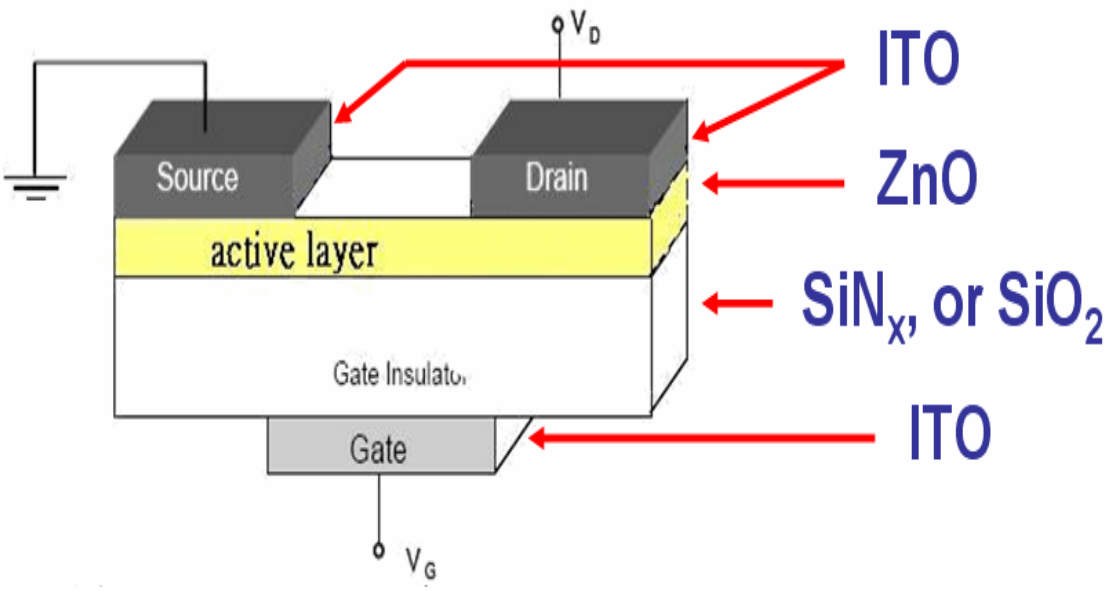

Figure 2-1 Assumed band structure of (a) un-doped and (b) Tin-doped In_2O_3. Shaded areas denote occupied states. Shift of the bands is apparent. Band-gaps, Fermi wave number (kF) and dispersion relations are also indicated

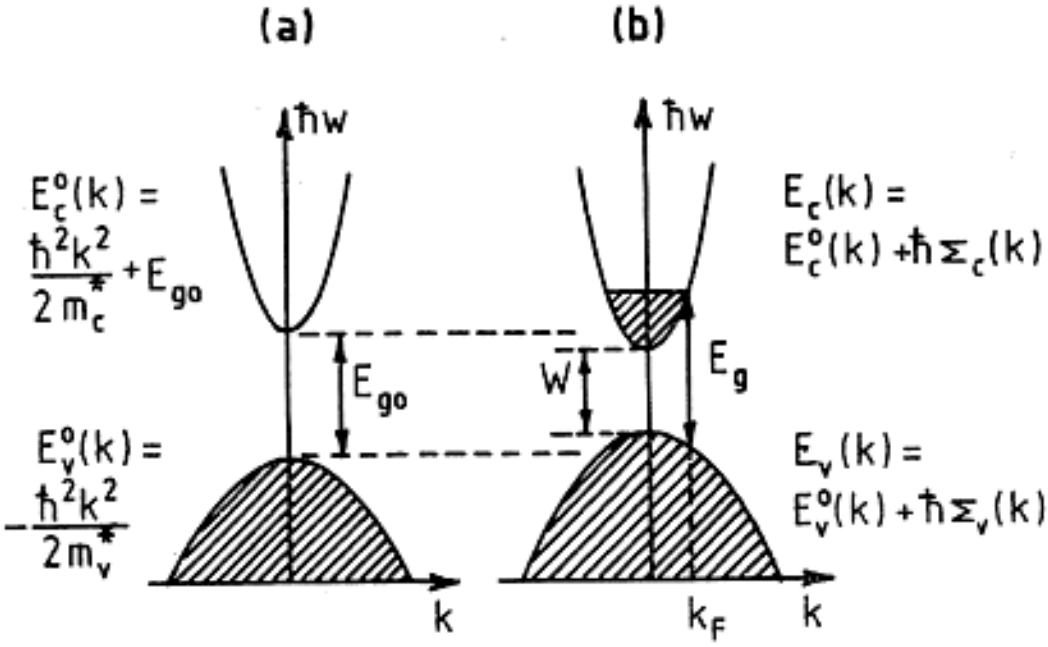

Figure 2(a) Schematically illustrated cross-sectional ZnO-based TFTs

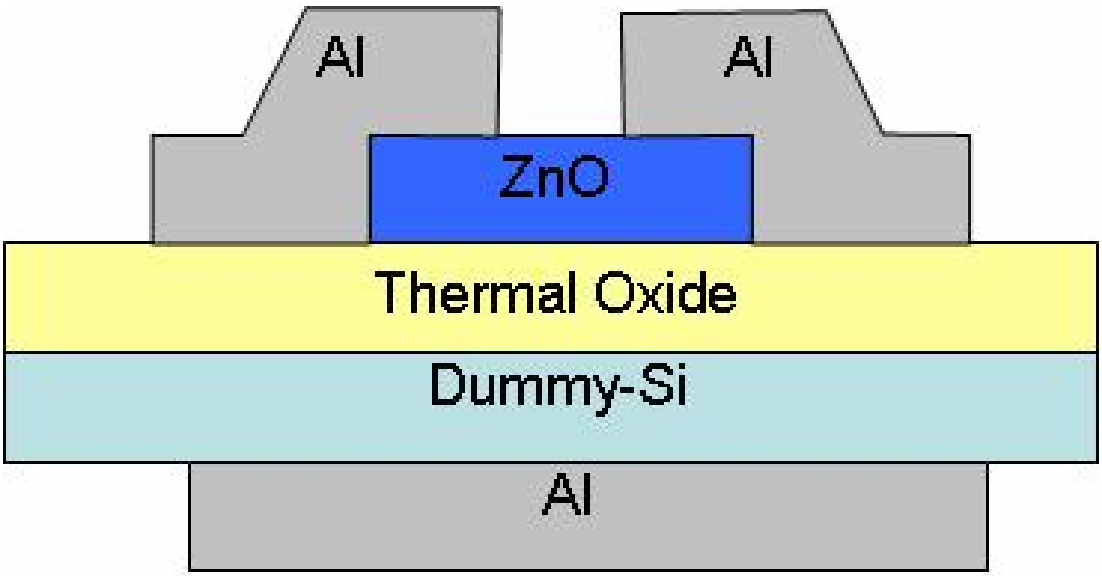

Figure 2(b) Schematically illustrated top-view ZnO-based TFTs

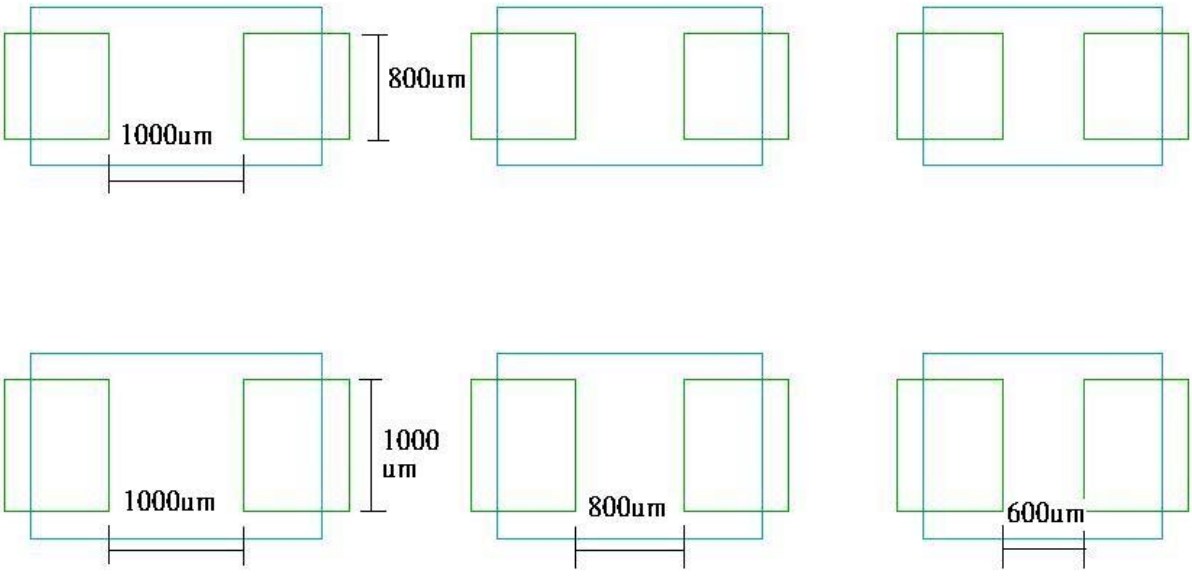

Figure 3 Schematic diagram of DC sputter system

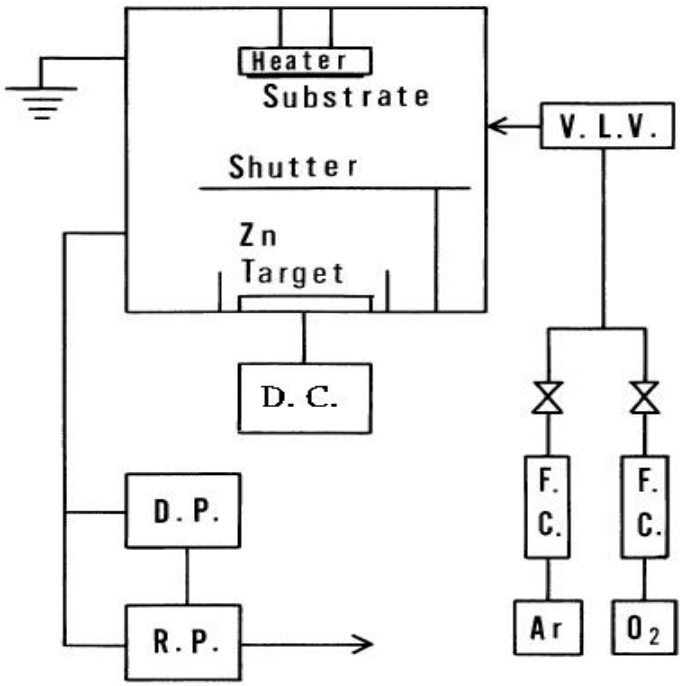

Figure 3-1 shows the resistance of different flow of oxygen

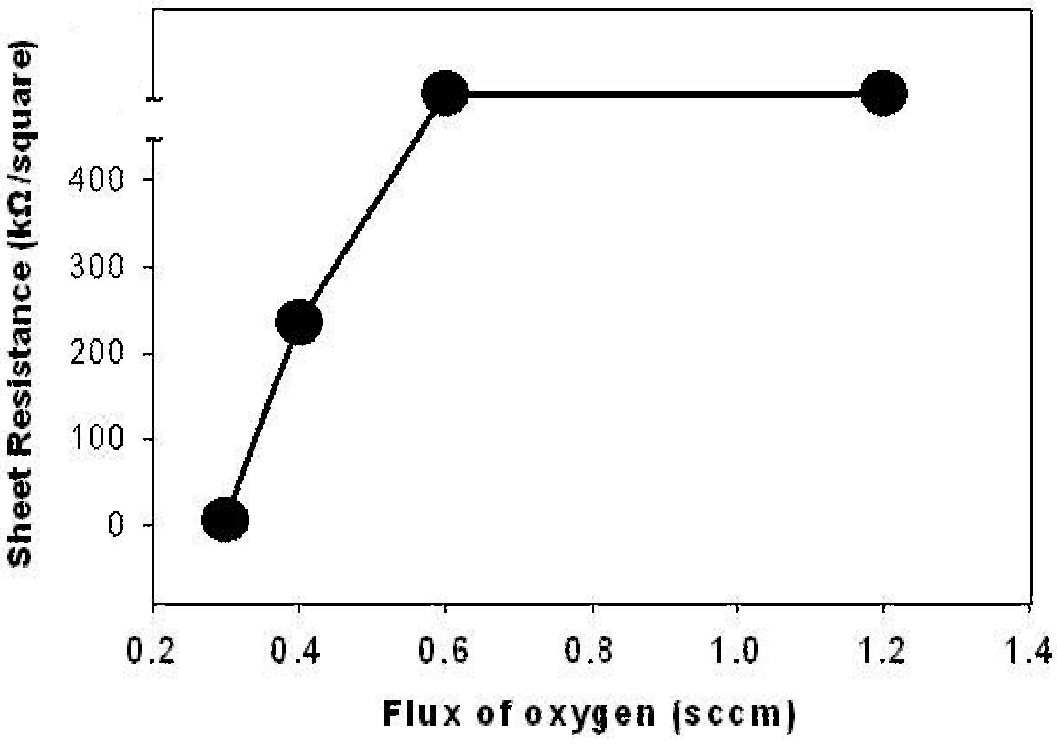

Figure 3-2 shows the relationship with different annealing temperature and resistance

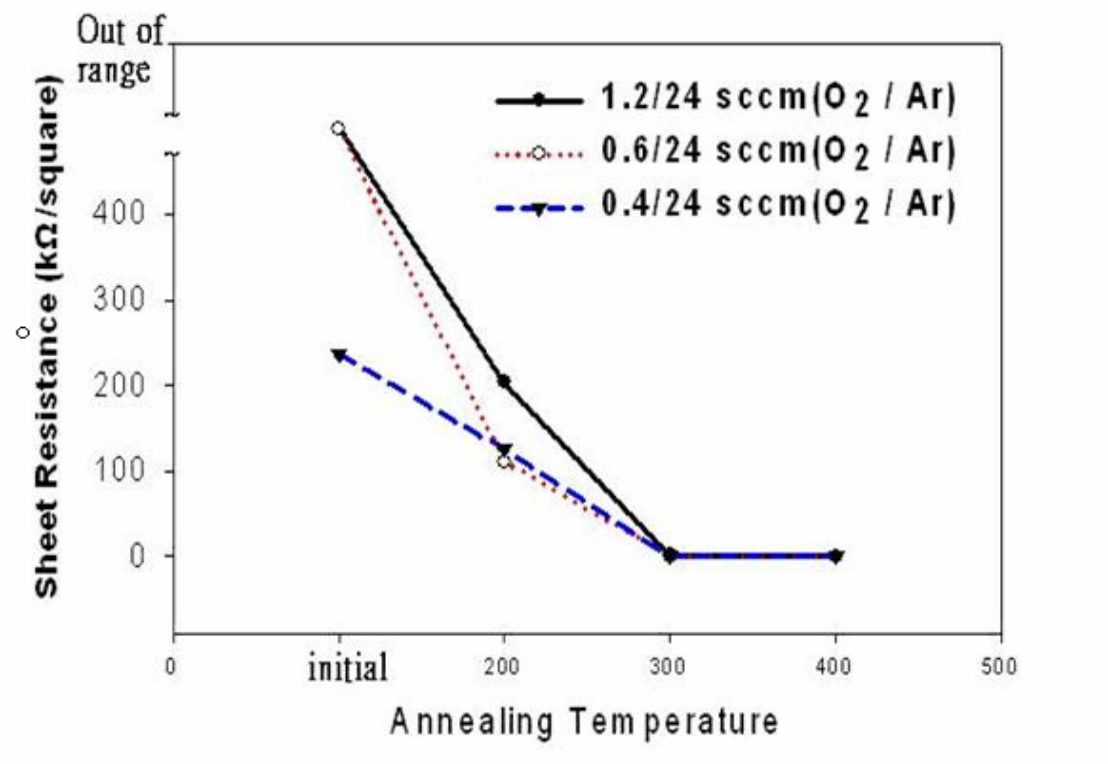

Figure 4 illustrates the measurement of as-deposited ZnO film on a single crystalline Silicon substrate (100) with different argon and oxygen mixture,

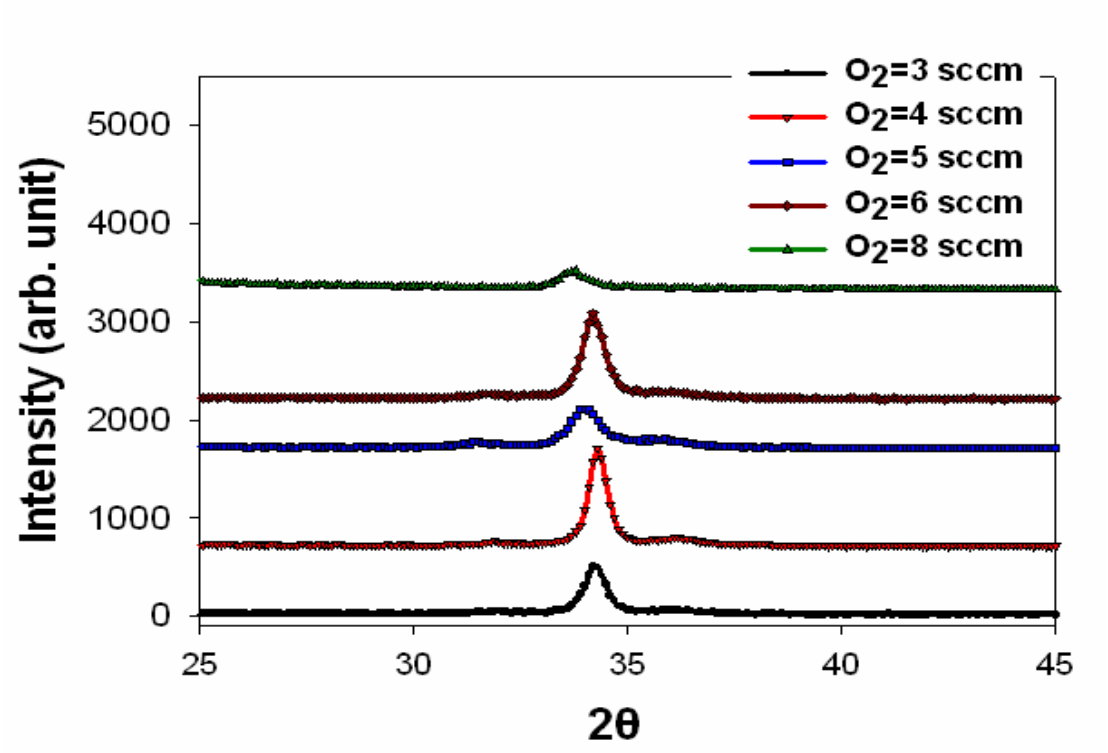

Figure 5 illustrates the measurement of (argon / oxygen) ratio (24 / 4) samples annealed in different temperature.

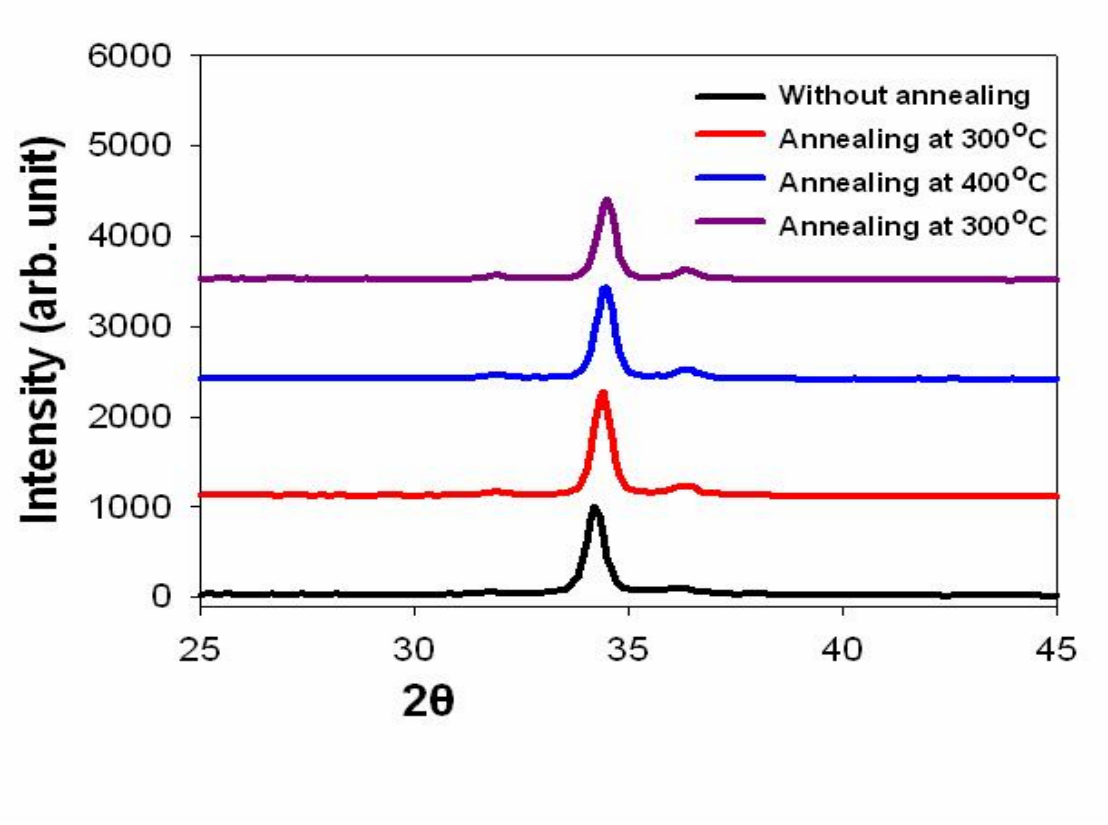

Figure 6 The comparison of different Argon / Oxygen rate in FTIR

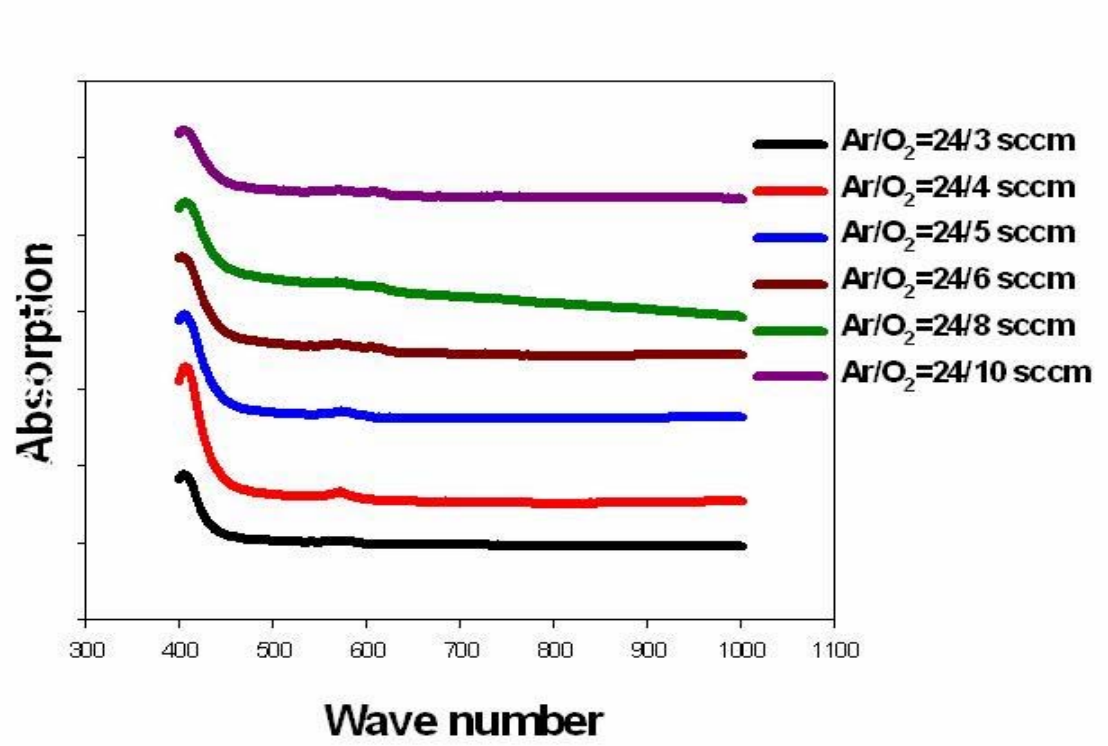

Figure 7 The comparison of different Argon / Oxygen rate in FTIR

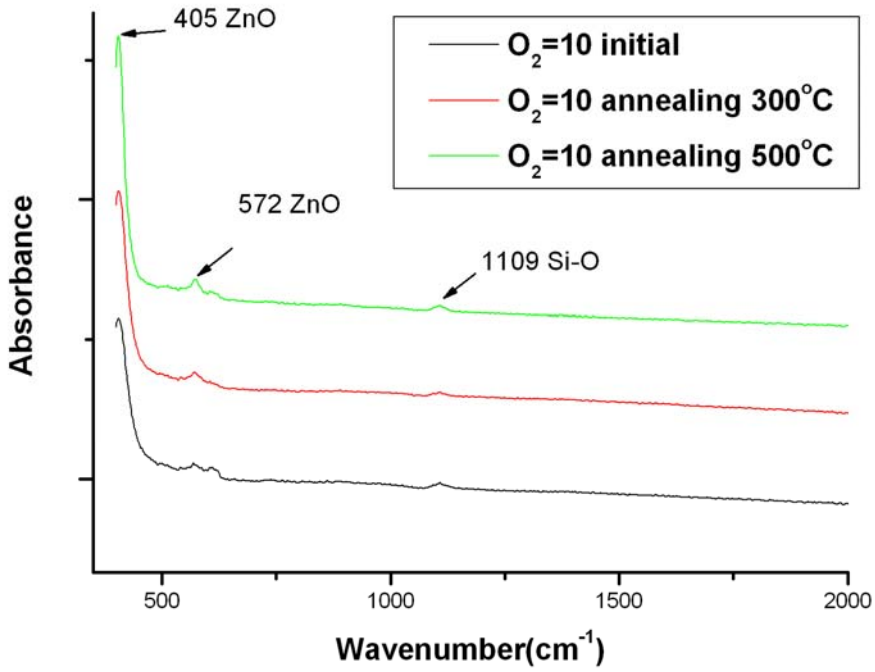

Figure 8 The I_D-V_G of our TTFTS in initial、300℃、500℃ annealing condition with Room Pressure Annealing Furnace.

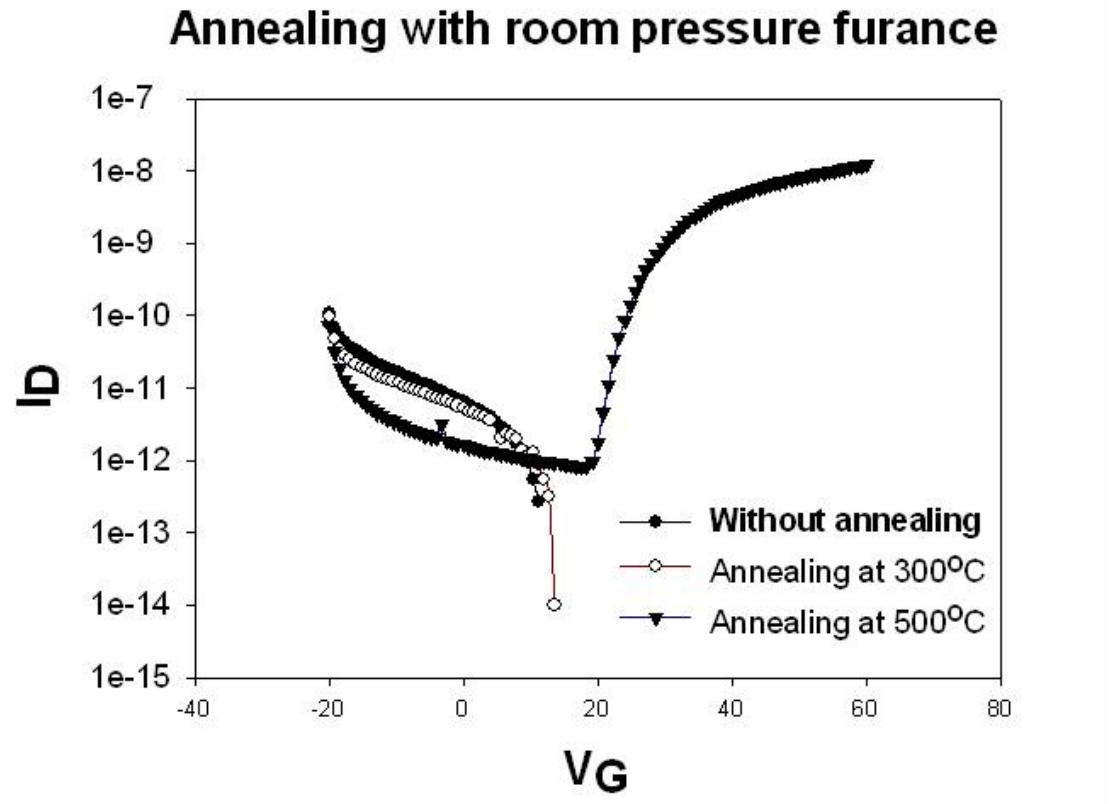

Figure 9 The I_D-V_D of our TTFTs in 500℃ with Room Pressure Annealing Furnace.

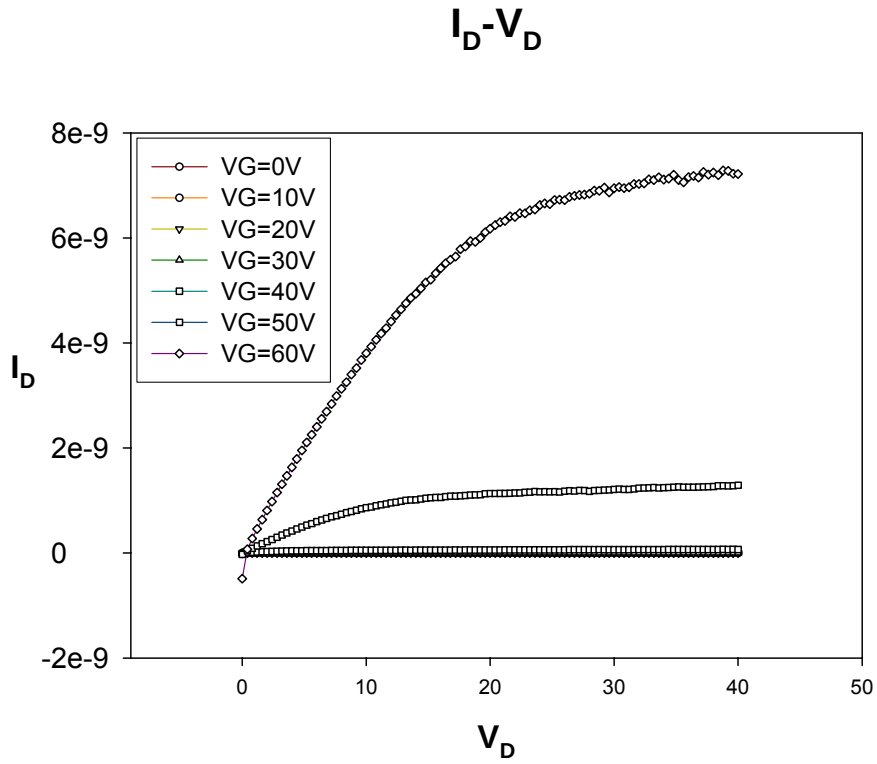

Figure 10 shows the I_D-V_G of the ZnO-based TFTs without annealing and in 300℃、400℃、500℃ with Vacuum Annealing Furnace annealing

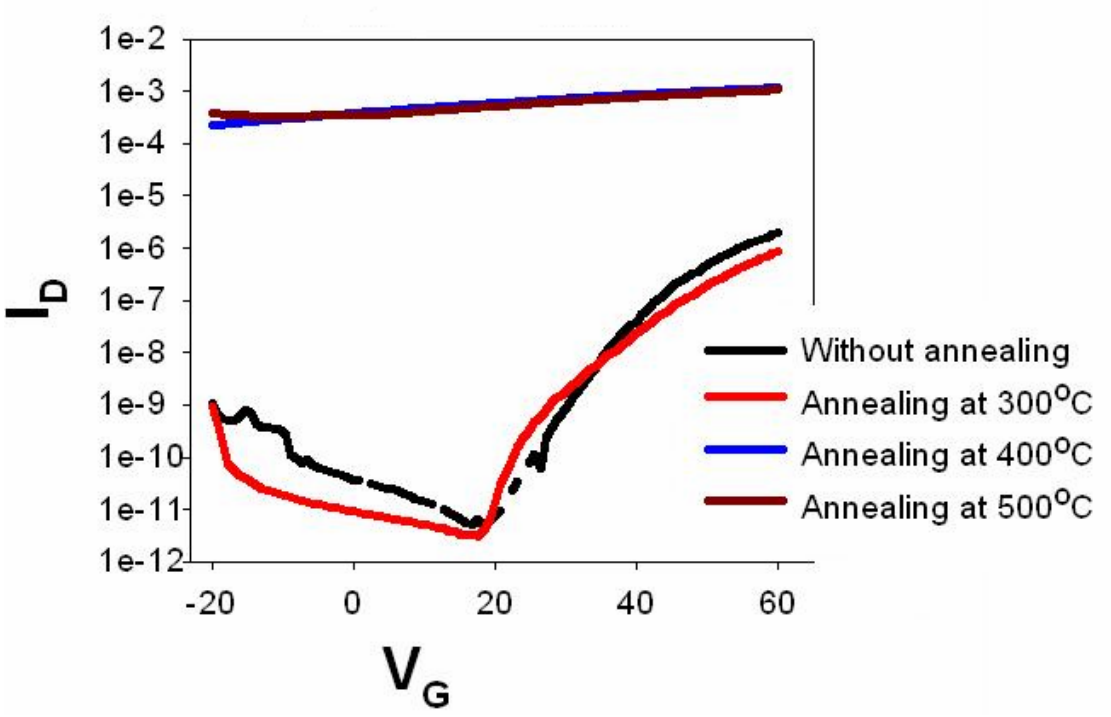

Figure 11 The I_D-V_D of our TTFTs in 300℃ annealing conditions

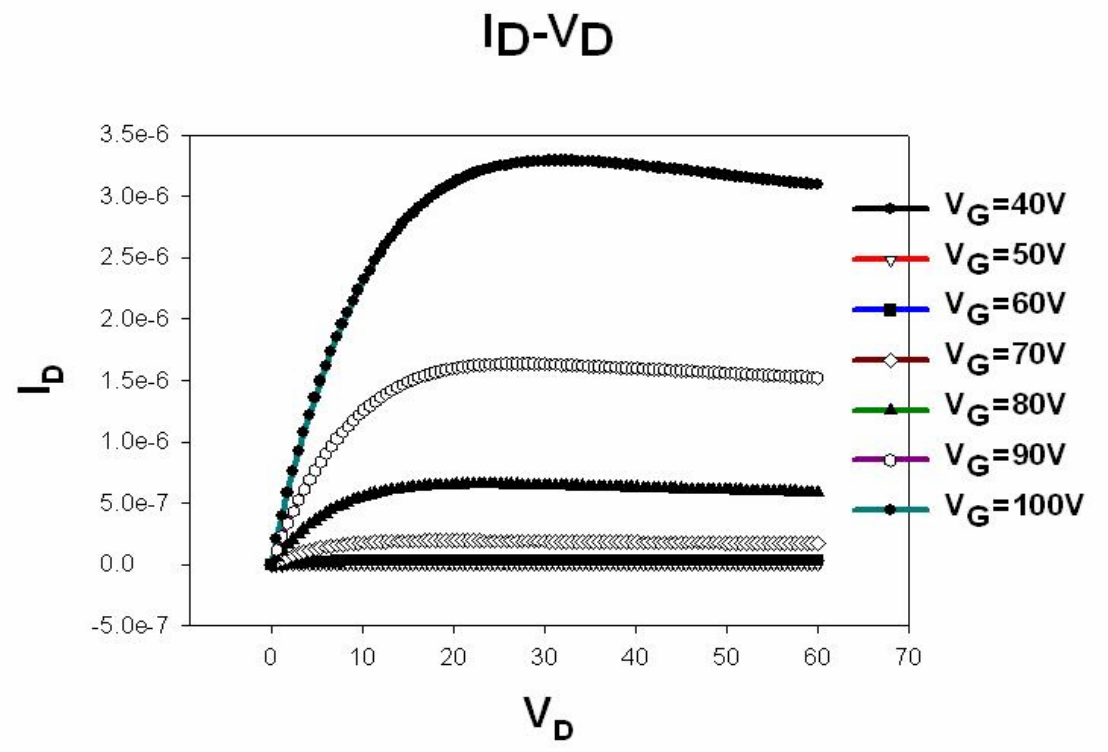

Figure 12 The I_D-V_G of the ZnO-based TFTs annealing in 0.05 mtorr, 0.1mtorr, 0.15 mtorr, and 0.2 mtorr with Vacuum Annealing Furnace.

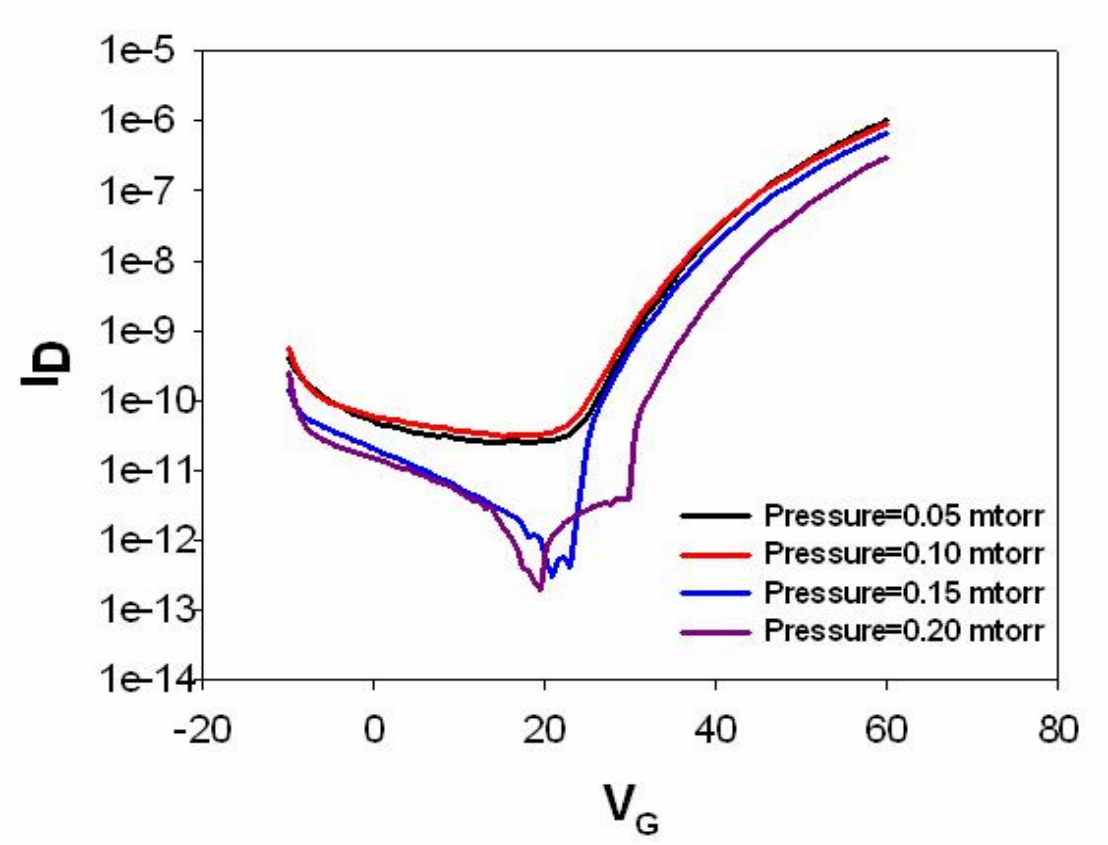

Figure 13 The relationship between annealing pressure and mobility change.

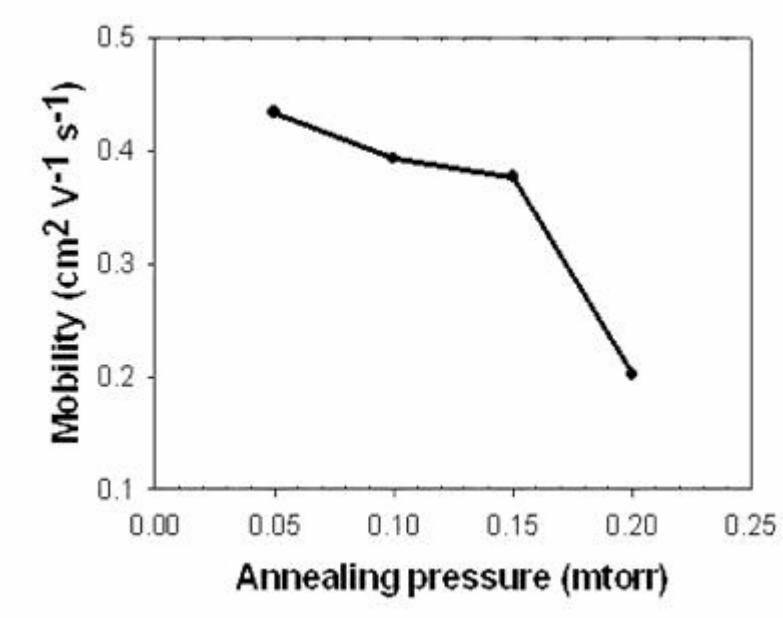

Table 1 There are some properties of Transparent Conducting Oxides at room temperature

Table 1.1 Some properties of transparent conducting oxides at room temperature.

Compound	Structure type	Cell dimensions (Å)			Resistivity (Ω cm)	Band-gap (eV)	Dielectric constant	Refractive index
		a	*b*	*c*				
SnO_2	Rutile	4.7371	—	3.1861	10^{-2}–10^{-4}	3.7–4.6	12 ($E\|\|a$) 9.4 ($E\|\|c$)	1.8–2.2
In_2O_3	C-rare earth	10.117	—	—	10^{-2}–10^{-4}	3.5–3.75	8.9	2.0–2.1
ITO	C-rare	10.117–10.31	—	—	10^{-3}–10^{-4}	3.5–4.6	—	1.8–2.1
Cd_2SnO_4	Sr_2PbO_4	5.5684	9.8871	3.1933	10^{-3}–10^{-4}	2.7–3.0	—	2.05–2.1
ZnO	Wurtzite	3.2426	—	5.1948	10^{-1}–10^{-4}	3.1–3.6	8.5	1.85–1.90

Table 2 Experimental Flow Path

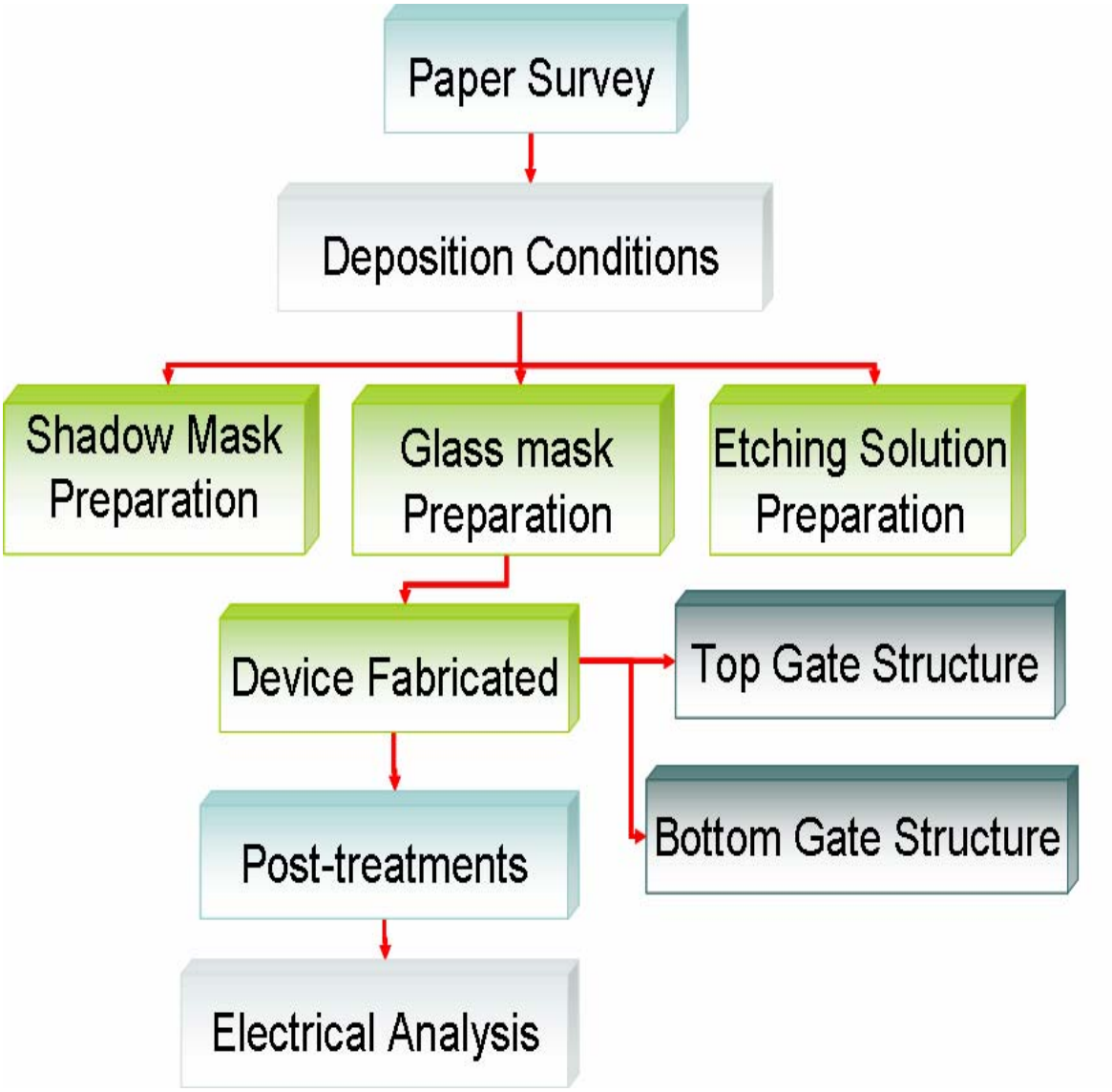

Table 3 Conditions of the DC power we applied

Pre-sputtering power	*Sputtering power*	*Uniformity*
60 W	100 W	Very bad
60 W	80 W	Bad
45 W	60 W	Good

Table 4 Experimental conditions of different (Argon / Oxygen) mixture ratio

Pre-sputter power	45 W					
Sputter power	60W					
Total pressure	7.6 × 10-3 Torr					
Substrate temperature	300 K					
Film thickness	1600 A					
Oxygen concentration Argon/Oxygen=24/x, x (Unit = sccm)	3	4	5	6	8	10

Table5 Etching solution list

H2O	HCl	HNO3	H3PO4	CH3COOH	Etching Time
4	1	0	0	0	＜1 sec
4	0	1	0	0	＜1 sec
9	1	0	0	0	＜1 sec
14	1	0	0	0	＜1 sec
200	20	3	0	0	＜1 sec
0	0	1	20	0	＜1 sec
200	20	3	0	3	1 sec
20	0	1	0	1	2 sec
20	0	1	0	1	2 sec
100	1	0	0	1	3 sec