Metal Oxides produced by Hollow Cathode Sputtering, their Utility in Thin Film Photovoltaics, and Results for AZO

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Abstract — An R&D-scale hollow cathode sputtering (HCS) system has been designed and built at NJIT. It operates in reactive mode using metal targets and can deposit virtually any metal oxide. We survey the use of metal oxide layers in thin film PV and report the preparation by HCS of several oxides including ZnO, ZnO:Al (AZO), Zn_xSn_yO , Cd_xSn_yO , and $Cu_{2.x}O$. Oxynitrides have also been prepared. For AZO, an excellent electron mobility of 53.4 cm²/Vs has so far been achieved, close to a world record for an as-deposited, sputter-based AZO layer. $Cu_{2.x}O$ layers have been successfully utilized as a back contact in CdTe solar cells. Work is under way to prepare the entire front stack of CdTe solar cells, namely TCO/HRT/buffer, by hollow cathode sputtering.

I. INTRODUCTION

Metal oxide layers can be employed in multiple roles in both thin-film and crystalline photovoltaic devices. These roles include: transparent conducting oxide, high resistance transparent layer, buffer layer, electron reflector, back optical reflector, and contact passivation [1, 2]. Hollow cathode sputtering offers several basic features that make it of interest for the preparation of such layers: low-cost metal targets, high deposition rate, repeatable processing, low-damage deposition, and the ability to be scaled up [3 - 5].

II. DEPOSITION SYSTEM AND LAYERS UNDER INVESTIGATION

We have recently built a hollow cathode deposition system at NJIT (Fig. 1). In its preferred reactive mode, oxygen is injected externally to the cathode and the high flow rate of argon prevents oxygen penetration into the cathode (Fig. 2). As a result, sputtering is conducted in metallic mode, i.e. the target surface is unoxidized. This in turn allows high process repeatability. The need to operate in transition mode (as required in conventional reactive sputtering) is avoided.

Two different cathodes were used in this work. Cathode #1 has an exit slot 6 cm in length, while that of cathode #2 is 15 cm in length. This dimension is perpendicular to the horizontal cross-section displayed in Fig. 2.



Fig. 1. Hollow cathode sputtering system at NJIT.



Fig. 2. Schematic of hollow cathode sputtering system (not to scale)

Examples of layers currently being pursued at NJIT are shown in Table I. It may be noted that mixed metal oxides, e.g. Zn_xSn_yO , can readily be prepared by HCS. Compound films prepared by related hollow cathode techniques at other laboratories include AZO [6], WO₃ [7], and CIGS [8].

TABLE I SOME HCS LAYERS CURRENTLY BEING PURSUED FOR PV APPLICATIONS AT NJIT

Layer	Material	Cell	Features
TCO	ZnO:Al	CIGS	High electron mobility, surface texture if desired
	Cd _x Sn _y O	CdTe	High performance, low cost
HRT	Zn _x Sn _y O	CdTe	Adjustable composition and band offsets
Window/ buffer	Zn _{1-x} Mg _x O:Al	CdTe	Adjustable composition and band offsets
Back contact	Cu _{2-x} O	CdTe	Adjustable composition, controlled Cu dose, electron back reflector

In superstrate thin-film CdTe solar cells the layers are sequenced as follows: glass/TCO/HRT/window-buffer/CdTe/ ohmic contact/metallization [9 - 12]. Since layers of Table I are produced by a reactive process, fine control of the oxygen partial pressure (and hence of the film transmission and resistivity) can be achieved by control of the oxygen flow rate. Other properties of interest for thin film PV include electron affinity and surface morphology. The electron affinity of mixed metal oxides can be modified through control of the metal ratio.

III. LASER REFLECTANCE MONITORING

We have employed laser reflectance monitoring to conduct basic growth rate studies for the case of ZnO deposition. In these experiments the beam from a 650 nm diode laser was reflected from a Si wafer mounted on the substrate holder and detected using a photodiode. In the HC sputtering configuration of Fig. 2, the Zn flux emanating from the cathode slot depends on the Ar flow rate. Figure 3 shows the photocurrent versus time for three different Ar flows. Assuming an excess of oxygen is always present, the ZnO growth rate is inversely proportional to the fringe period.



Fig. 3. Laser reflectance monitoring during AZO growth with three different argon flows.

Figure 4 summarizes the growth rate data obtained by this method. The growth rate rises with increasing Ar flow and finally saturates once the majority of the sputtered Zn atoms can be removed from the cathode cavity without re-depositing on the targets. This data was obtained using cathode #1.



Fig. 4. Relative deposition rates as a function of Ar flow as determined by laser reflectance monitoring.

In the neighborhood of oxygen flows suitable for AZO growth it was found that higher oxygen flows led to slightly enhanced growth rates (see Table II). Since the Zn flux is fixed, and the film stoichiometry is still closely that of Zn:O = 1:1 this implies that at the lower oxygen flows some Zn must be desorbed from the growing film without forming ZnO. This is not unreasonable as the vapor pressure of Zn at 500K is 3.6×10^{-3} Pa.

 TABLE II

 INFLUENCE OF O_2 FLOW RATE ON GROWTH RATE

Ar flow rate (sccm)	O ₂ flow rate (sccm)	Fringe period (s)	Relative growth rate
1150	12.5	403	1.00
1150	17.0	356	1.13

IV. RESULTS FOR AZO AND ZNO

Aluminum-doped ZnO (AZO) is often used as the top TCO for $Cu(In, Ga)Se_2$ solar cells. Large scale production of high quality AZO in the thickness required for low sheet resistance is a major cost element for CIGS manufacturing.

Thin-film, polycrystalline AZO was produced by HCS using metallic Zn:Al alloy targets. The substrates were 7.5 cm x 5.0 cm SLG microscope slides 1 mm in thickness. The typical optical properties of AZO films produced by HCS are shown in Fig. 5. The carrier concentration *n* for the film produced in run 153 was 4.0×10^{20} /cm³ while that for run 8 was smaller

by a factor of about two. A slight blue shift in the absorption edge for the former film due to the Burstein-Moss effect is evident, as is the higher plasma frequency $\omega_p = (ne^2 / \varepsilon_0 \varepsilon_\infty m_e^*)^{1/2}$.



Fig. 5. Spectral transmittance and reflectance of AZO films.

As is well known, it is easy to control the doping level of AZO via Al content but not so easy to produce films with high electron mobility μ . A high electron mobility is important for a TCO since for a given sheet resistance R_{sh} the visible absorptance varies as $1/\mu^2$ [1].

In order to showcase the capabilities of HCS we chose first to explore the optimization of AZO with the goal of reaching a record mobility. Over 100 runs were conducted and all of the basic deposition parameters were varied to uncover the most favorable conditions. Figure 6, for example, shows the effect of substrate temperature on Hall mobility. These early studies were conducted using cathode #1 operated with DC sputtering power.



Fig. 6. Electron mobility of AZO versus substrate temperature for runs in which all other parameters were identical.

In another study, the effect of varying the oxygen flow around its optimal value (as regards mobility) on film carrier concentration, mobility, and thickness (for fixed deposition time) was determined (see Fig. 7). It was discovered that increasing the oxygen flow reduces n and tends to increase the film thickness (as confirmed by laser reflectance monitoring).



Fig. 7. Measured AZO parameters (n, t, μ) versus oxygen flow.

Subsequent optimization studies investigated the effect of deposition pressure, film thickness, throw distance, a comparison of DC versus mid-frequency pulsed DC with a reverse off voltage, and cathode #1 versus cathode #2. The parameters of the best AZO films produced to date are shown in Table III. The Al-free films are discussed in section V.

TABLE III PARAMETERS FOR THE HIGHEST MOBILITY AZO AND ZNO FILMS PREPARED BY HCS TO DATE

Matl.	t nm	<i>R_{sh}</i> Ω/sq.	ho Ω cm	n _e /cm ³	μ _H cm²/Vs
			Cathode #1		
ZnO:Al	952	3.45	3.28 x 10 ⁻⁴	4.06 x 10 ²⁰	46.9
			Cathode #2		
ZnO:Al	1338	2.16	2.89 x 10 ⁻⁴	4.05 x 10 ²⁰	53.4
ZnO	841	-	1.09 x 10 ⁻³	1.39 x 10 ¹⁹	41.2
ZnO:H	743	-	2.83 x 10 ⁻³	3.96 x 10 ¹⁹	66.3

To put our mobility results in context we surveyed 60 published papers on AZO prepared by sputtering and noted the maximum reported mobility in each paper. Some of the more interesting papers are cited in references [13 - 24]. These maximum mobilities for as-deposited films ranged from 7.5 cm²/Vs to 57.0 cm²/Vs with a median value of 28 cm²/Vs. The film with $\mu = 57$ cm²/Vs [22] was the sole sputtered AZO film found in the literature with a higher mobility (obtained with the benefit of a relatively low *n* of 2.6 x 10²⁰ /cm³) than our record AZO film with $\mu = 53.4$ cm²/Vs.

To verify process reproducibility, a run was conducted 9 runs after achieving a good mobility result using identical settings and flows. The measured properties of these two films are shown in Table IV. To within experimental accuracy, the film properties were almost identical.

TABLE IV						
REPRODUCIBILITY CHECK						
Matl.	t nm	<i>ρ</i> Ω cm	n _e /cm ³	μ _H cm²/Vs		
ZnO:Al	1300	2.84 x 10 ⁻⁴	4.20 x 10 ²⁰	52.3		
ZnO:Al	1340	2.87 x 10 ⁻⁴	4.10 x 10 ²⁰	53.1		

V. MODELING OF MOBILITY

Mobility is reduced by various electron scattering mechanisms, the most important of which for degenerate AZO films is ionized impurity scattering. Hence the upper limit for mobility tends to decrease as the carrier concentration increases. The maximum mobility μ_{ii} allowed by ionized impurity scattering is given by

$$\mu_{ii} = \frac{3(\varepsilon_0 \varepsilon_r)^2 h^3}{Z^2 m_e^{*2} e^3} \frac{n_e}{n_{ii}} \frac{1}{F_i(\xi)}$$
(1)
$$F_i(\xi) = \ln(1+\xi) - \frac{\xi}{1+\xi} \qquad \xi = (3\pi^2)^{1/3} \frac{\varepsilon_0 \varepsilon_r h^2 n_e^{1/3}}{m_e^{*} e^2}$$

where ε_0 is the permittivity of free space, ε_r is the relative static permittivity, *h* is Planck's constant, and $n_e (= N_D - N_A)$ is the free electron concentration; *Z* and n_{ii} are the charge and concentration of the impurities, respectively [1]. In ZnO, the conduction band is non-parabolic [25], and taking this into account we have calculated μ_{ii} as a function of n_e . This is shown in Fig. 8 as the red dot-dash curve. Nevertheless, there are some uncertainties in this result as conflicting equations are given in the literature and the values of certain parameters have to be assumed.



Fig. 8. Electron mobility of ZnO versus carrier concentration: solid curve for single-crystal ZnO according to the Masetti formula;

dashed curve for polycrystalline ZnO taking into account the barrier at grain boundaries; dot-dash curve ionized impurity scattering alone.

An alternative approach to predicting the general behavior of $\mu(n_e)$ is to adopt the empirical Masetti formula widely used for describing the mobility in single-crystal Si [26]. This was done by Ellmer [19]. With a choice of parameters somewhat different from those adopted by Ellmer we have calculated the electron mobility μ_n for single-crystal ZnO according to the Masetti formula

$$\mu_{n} = \mu_{0} + \frac{\mu_{\max} - \mu_{0}}{1 + \left(\frac{n}{n_{1}}\right)^{\alpha}} - \frac{\mu_{1}}{1 + \left(\frac{n_{2}}{n}\right)^{\beta}}$$
(2)

The result is shown as the solid curve in Fig. 8. At low *n* the mobility plateaus at a value determined by lattice scattering (about 210 cm²/Vs). As *n* increases the mobility falls because of ionized impurity scattering while at high dopant concentrations the dopants may form clusters with higher effective charge *Z* that strongly reduces the mobility.

To account for the polycrystalline nature of the AZO films we include the need for the carriers to overcome a barrier E_b in the conduction bands at the grain boundaries yielding an effective mobility μ_{eff} given by

$$\mu_{eff} = \mu_0 \exp\left(-\frac{E_b}{kT}\right) \tag{3}$$

where E_b varies inversely with *n* and the prefactor μ_0 is interpreted as the mobility inside the grain [1]. By combining the mobilities given by equations (2) and (3) we calculate the dashed curve in Fig. 8 for polycrystalline ZnO. The position of the LH side of this curve is dependent on the assumed values of trap density at the grain boundary, grain size, etc. These parameters were chosen to yield a $\mu(n)$ curve consistent with the experimental data for the best NJIT films. Figure 9 reproduces this $\mu(n)$ curve on an expanded scale.



Fig. 9. Expanded view of the modeled dashed curve from Fig. 8. Data points are for the highest mobilities at a given concentration from Hall measurements on ZnO and AZO films prepared at NJIT by

HCS and on the best films reported in the literature.

The plot in Fig. 9 also shows data points for (μ, n) pairs for films having the highest mobilities uncovered in our literature search, for NJIT AZO produced by HCS (cathodes #1 and #2), and for NJIT ZnO produced by HCS using pure Zn targets. The blue dashed curve is seen to fairly accurately describe the upper limit of experimental mobility for thin film ZnO across a wide range of carrier concentrations.

With Al-free ZnO films (solid squares in Fig. 9) we achieved remarkable mobilities up to 66.3 cm²/Vs (see Table III). We believe these results in the range $10^{19} < n < 10^{20}$ /cm³ are a notable achievement among all thin film ZnO and especially for sputtered films. For $n \approx \text{mid}-10^{16}$ /cm³ higher mobilities have been reported e.g. $\mu > 140 \text{ cm}^2$ /Vs for ZnO epitaxially grown on *c*-plane sapphire at 750°C [27] and $\mu = 72 \text{ cm}^2$ /Vs for $n \approx \text{mid}-10^{17}$ /cm³ [28].

We do not yet know for certain the structural factors that allow the observed high mobilities. A preliminary SEM crosssection (Fig. 10) did not exhibit the anticipated columnar structure but appeared featureless. Two comments can be made. Firstly, although atoms of the target material are sputtered and sputtering is generally classified as a physical vapor deposition process, in HCS the sputtered atoms (and the reactive oxygen species) are transported to the substrate in a flow of argon at the relatively high pressure of ~ 250 mTorr. At this pressure the mean free path $\lambda = kT/\sqrt{2\pi d^2 P}$ is approximately 0.2 mm. Thus, it would appear that the actual deposition process of HCS is more like CVD (chemical vapor deposition) than PVD. Secondly, at these pressures energetic particles are thermalized and there is no lattice damage from particle bombardment.



Fig. 10. SEM micrograph of fracture cross-section of AZO on glass prepared by HCS (film on right-hand side).

VI. RESULTS FOR OTHER MATERIALS

Other materials that we have prepared by HCS include ZnO_xN_y (zinc oxynitride), a layer suitable for TFT fabrication,

 Cd_xSn_yO (cadmium tin oxide) which we are developing as a TCO for CdTe solar cells, and $Cu_{2-x}O$ as an ohmic contact. We have prepared $Cu_{2-x}O$ of different composition and band gap, and have achieved cell efficiencies comparable to those previously obtained using evaporated Cu and with higher reproducibility. Some preliminary properties of ZnO_xN_y and Cd_xSn_yO are shown in Table V. A mobility of 41.6 cm²/Vs was achieved for a Cd_xSn_yO layer annealed under a glass/CdS cap.

 $TABLE \ V$ Parameters for the Highest Mobility ZnO_xN_y and Cd_xSn_yO Films Prepared by HCS to Date

Matl.	t nm	<i>R_{sh}</i> Ω/sq.	<i>ρ</i> Ω cm	n _e /cm ³	μ_H cm²/Vs	
Cathode #1						
ZnO _x N _y	465	-	14.6	2.17 x 10 ¹⁶	19.6	
Cd _x Sn _y O	414	8.91	3.69 x 10 ⁻⁴	5.27x 10 ²⁰	32.2	
annealed	344	10.1	3.49 x 10 ⁻⁴	4.29x 10 ²⁰	41.6	

VII. CONCLUSIONS

Linear hollow cathodes of a new design were fabricated and have been used to prepare functional metal oxide, mixed oxide, and oxy-nitride layers by hollow cathode sputtering. We have produced AZO films with near world record mobilities for as-deposited, sputtered films. ZnO films of lower carrier concentration have been produced with mobilities up to 66.3 cm²/Vs.

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