Effect of self-grown seed layer on thermoelectric properties of ZnO thin films

S. Saini^{1, †}, P. Mele^{2, †}, H. Honda³, T. Suzuki³, K. Matsumoto⁴, K. Miyazaki⁵, A. Ichinose⁶, L. Molina Luna⁷, R. Carlini⁸, and A. Tiwari¹

¹ Department of Materials Science and Engineering, University of Utah, Salt Lake City, Utah, USA

² Research Center for Environmentally Friendly Materials Engineering, Muroran Institute of Technology, Muroran, JAPAN,

³ Department of Quantum matter, ADSM, Hiroshima University, Higashi-Hiroshima, JAPAN

⁴ Department of Materials Science, Kyushu Institute of Technology, Kitakyushu, JAPAN

⁵ Department of Mechanical Engineering, Kyushu Institute of Technology, Kitakyushu, JAPAN

⁶ Electric Power Engineering Laboratory, Central Research Institute of Electric Power Industry (CRIEPI), Yokosuka, JAPAN

⁷ Department of Materials and Geosciences, Technical University of Darmstadt, Darmstadt, GERMANY

⁸ Department of Chemistry and Industrial Chemistry, University of Genova, ITALY

Email: ssaini@mse.utah.edu , pmele@mmm.muroran-it.ac.jp

[†] Authors contributed equally to this work

Abstract:

The influence of self-grown seed has been studied on thermoelectric performance of 2% Al-doped ZnO (AZO) thin films. The thickness and orientation of *c*-axis domains in seed layer change on different substrates while other deposition conditions were kept unchanged for a comparable study. The changes occur because of different nucleation process of thin film growth on substrate interface and the different lattice mismatch between AZO and substrate. Thin films are grown by pulsed laser deposition on single crystals (SrTiO₃ (STO) and Al₂O₃) and cheap amorphous fused silica (FS) substrates at 400°C. All thin films are *c*-axis oriented. The grains are highly connected and elongated in shape which leads to high thermoelectric properties. The thicker self-grown seed layer is found in thin film deposited on FS substrates which shows best performance: electrical conductivity $\sigma = 93 \text{ S/cm}$ and Seebeck coefficient $S = -203 \text{ } \mu\text{V/K}$, which estimate power factor ($S^2 \cdot \sigma$) about $0.37 \times 10^{-3} \text{ Wm}^{-1}\text{K}^{-2}$ at 600 K. The value of thermal conductivity (κ) was found lowest ($4.89 \text{ Wm}^{-1}\text{K}^{-1}$) for thin film deposited on FS than the other thin films ($6.9 \text{ Wm}^{-1}\text{K}^{-1}$ on Al₂O₃ and $6.55 \text{ Wm}^{-1}\text{K}^{-1}$ on STO) at 300 K. The figure of merit, $ZT = S^2 \cdot \sigma \cdot T / \kappa$, is calculated 0.046 at 600K, 5 times larger than the ZT of our previous reported bulk AZO, which is promising for practical applications of thermoelectric oxide thin films.

Keywords: Self-grown seed layer, *c*-axis orientation, Al-doped ZnO thin films, pulsed laser deposition, Seebeck coefficient, high temperature applications, thermoelectric oxides, figure of merit

1. Introduction:

ZnO is an n-type semiconductor with versatile applications such as optical devices in ultraviolet region [1], piezoelectric transducers [2], transparent electrode for solar cells [3], gas sensors [4]. Apart from it, ZnO is also a good candidate for thermoelectric applications [5, 6] since it is of low-cost, nontoxic, and stable over a wide range of temperature. Several reports were published on sintered ZnO added with various different dopants in order to improve its thermoelectric performance. Comprehensive review on bulk ZnO thermoelectric can be found in [7]. Thin films are advantageous over bulk materials because of their light weight, quick response time, and compact size for module or sensors. In perspective, tiny and flexible modules based on oxide thin films may be used to convert in energy the waste heat generated by car engines, power plants, steel furnace (temperatures 1000 K or more) [8]. That is why in recent years, many research groups have intensively investigated on the growth of high quality ZnO thin films for various applications [9-12]. Thermoelectric ZnO thin films are not enough explored, even if they are relatively easy to prepare [13-19]. The main reason is the practical difficulty in the measurement of thermoelectric properties at high temperature.

Our previous works [20-23] demonstrate the influence of deposition temperature on thermoelectric performance of AZO thin films. The change in deposition temperature leads to change in grain size and orientation which, in turn, leads to change in thermoelectric performance. In this study, we are extending our previous work and reporting the influence of self-grown seed layer at substrate-thin film interface on thermoelectric performance of 2% Al-doped ZnO (AZO) thin films. The growth of this seed layer during deposition in pulsed laser deposition (PLD) technique starts with nucleation process when the AZO plasma plume reaches

on substrates. Due to the lattice mismatch, the strain energy is released from the interface during the nucleation process. Two cases are possible here. If the strain energy is large due to a big mismatch between substrate and film, the nucleation process starts with growth of islands forming a 3D-cluster seed layer; after reaching a critical thickness of the 3D clusters, strain is released then the film continue to grow with layer-by layer mechanism. The second case occurs when the strain energy is moderate due to a small mismatch: the layer-by-layer growth starts directly on the substrates [24]. In this work, we will discuss how seed layer is formed depending on substrate, influencing the thermoelectric properties of AZO films.

2. Experimental details:

The AZO thin films were grown by PLD technique using Nd:YAG laser (266 nm, 10 Hz). Pellet of $\text{Zn}_{0.98}\text{Al}_{0.02}\text{O}$ (20 mm in diameter and 3 mm in thickness) prepared by spark plasma sintering was used as target to grow the thin films. A detailed description of sintered target preparation is reported elsewhere [25]. The laser was shot on the dense AZO target with an energy density of about 4.2 J/cm^2 for deposition period of 30 min. Thin films were deposited on SrTiO_3 100 (STO), Al_2O_3 (0001) single crystal and low cost fused silica substrates at 400°C under an oxygen pressure of 27 Pa. The target was rotated during the irradiation of laser beam. The STO substrates were glued with silver paste on inconel plate customized for ultrahigh vacuum applications. The thickness of thin films was kept about 500 nm. Deposition parameters such as pulse frequency (10 Hz), substrate-target distance (about 35 mm) and rotation speed of the target (25 rotations per minute) were kept unchanged during all the deposition routines.

The structural characterization was performed by X-ray diffraction (at wavelength of $\text{CuK}_\alpha = 1.54056 \text{ \AA}$) (XRD) (Bruker D8 Discover) in θ - 2θ configuration and morphology was

checked by scanning electron microscope (SEM; JEOL, FESEM) at accelerating voltage of about 15 kV and transmission electron microscopy (TEM; JEOL 2010F) at accelerating voltage of 160 kV. TEM specimens were thinned out by mechanical grinding, polishing, and dimpling. This process is followed by Ar-ion milling. The thickness and in-plane roughness were obtained by a Keyence VK-9700 3D microscope. The electrical conductivity was measured from 300 -600 K by a custom-built four-point-probe technique consisting of a current source (ADCMT 6144), a temperature controller (Cryo-con 32) and a nano voltmeter (Keithley 2182A). Seebeck coefficient were measured from 300 -600 K with a commercially available system (MMR Technologies SB-100). It should be noted that 600 K is the maximum limit of temperature for the available instrumentation. Carrier concentrations at room temperature were evaluated by means of Quantum Design PPMS. The thermal conductivities of thin films at room temperature were measured with time domain thermoreflectance [26, 27].

3. Results and discussion:

Figure 1(a) shows XRD pattern of AZO thin film deposited on single crystal STO, Al_2O_3 and amorphous silica substrates at 400°C. These films are *c*-axis oriented and the secondary phase such as ZnAl_2O_4 did not form during the deposition process. The *c*-axis length is shrunk for thin films deposited on Al_2O_3 (5.19Å), FS (5.19Å) and same as bulk AZO (5.20Å) for thin films deposited on STO (5.20Å). The ϕ -scan (figure 1(b)) gives 12 peaks which indicates mixed structure with 30° rotated and non-rotated hexagonal AZO unit cells oriented along *c*-axis of STO. For thin films AZO/ Al_2O_3 gives 6-fold symmetry which is the sign of epitaxial growth. There are no ϕ -scan peaks found for the thin film deposited on amorphous FS substrates.

The effect of substrates on morphology of thin films is studied by SEM images. Top-view

SEM images of thin films AZO/STO, AZO/ Al_2O_3 and AZO/FS are shown in figure 2. Thin films are highly connected and the size of grains is almost same for thin films grown on single crystal. The grains of thin films are bigger on amorphous substrates (90 nm) than single crystal (60 nm) substrates. We found flaks like grains for thin films on amorphous FS substrates and rounded (AZO/STO) or elongated (AZO/ Al_2O_3) on single crystal substrates which can be directly related to the crystal structure of substrates. This kind of growth on the single crystals reduce the possibility to grow spikes on surface which results smooth surface by layer formation during the growth process [28]. We notice that the surface of thin film on single crystal is smoother than amorphous substrates as shown in figure 2. Conversely, the nucleation of thin film atom cluster (3D-growth) on amorphous FS substrate leads to higher roughness than the film deposited on single crystal substrates.

Figure 3 shows cross-sectional TEM image of thin films deposited on single crystal and amorphous substrates. Grains are highly connected and vertically aligned with a typical columnar growth as shown in figures 3 (a, d, and g) for thin films deposited on different substrates. Magnified images of interface of substrate and thin films are shown in figures 3 (b, e, and h). It can be easily seen at interface that thin film deposited on FS presents a self-grown seed layer which is absent in thin films grown on STO. The thickness of self-grown seed layer is lesser on Al_2O_3 (40 nm) than the thin film on FS (100 nm). Magnified image of the thin film and substrate interface shows layers of bunch of unit cells. The layers are clear and uniformly distributed in thin film AZO/STO shown in figure 3(c). Thin film AZO/ Al_2O_3 shows layers in different orientation but the domain are bigger for these layers (circle in figure 3(f)). Layers are randomly oriented for thin films AZO/FS (figure 3 (i)).

The column growth shows spikes structures in thin films grown on FS [21]. However

these spikes structures are missing in thin films grown on single crystal substrates [20]. These spike structures are the result of film-atom cluster growth mode (3D-growth) of thin films because of completely different morphology of substrate and thin films lattice parameters. In the case of STO, the lattice mismatch with AZO is moderate: -2% in *c*-axis direction and -6% on *a*-axis direction. Consequently, the AZO layers is able to grow epitaxially on the *c*-axis direction directly on the substrate following a layer-growth mechanism and elongated grains parallel to *c*-axis (and occasionally *a*-axis oriented grains) may form easily due to less strain energy. The seed layer appear in AZO/Al₂O₃ thin film and the thickness is very less about 40 nm (figure 3 (e)). The formation of seed layer may be attributed to the larger mismatch between Al₂O₃ substrate and AZO layer: 19% along *c*-axis direction. In order to partially release the stress, randomly oriented grains are formed at first. On amorphous substrates (fused silica) it is not possible to calculate mismatch, however larger strain energy is expected and to release it the formation of thicker random oriented layer is reasonable. This argument may be validated in near future by advanced observations as Geometrical Phase analysis [29].

Transport and thermoelectrical characterization is shown in figure 4 in the range of 300 K to 600 K and reported in table 1. Figure 4 (a) shows inverse temperature dependence of natural logarithmic function of conductivity ($\ln(\sigma T)$ vs $1000/T$). The linearity of the plots indicates that thermally activated hopping mechanism is possible in electrical conductivity. The electrical conductivity follows relation $\sigma T = A \exp(-E_\sigma/kT)$ (where E_σ , the activation energy for electrical conductivity; A , pre-exponential factor and considered as temperature independent; k , Boltzmann constant; σ , electrical conductivity; T , absolute temperature) [28]. The value of activation energy E_σ is calculated by the slope of linear fit of the plots and belongs to the range of 3 to 6 meV. σ increases with temperature, with a typical semi conducting behavior. Electrical conductivity is

higher for thin film AZO/Al₂O₃ for all temperature range than thin films AZO/STO, AZO/FS as shown in figure 4(a) and table 1. This can be explained by micro-structure of the thin films. The grains of thin films AZO/Al₂O₃ are elongated (figure 2) whereas thin films AZO/STO have rounded grains and thin film AZO/FS has flakes type of grains. As a result, boundary scattering of charge carrier is reduced in the thin film AZO/Al₂O₃ and, hence, its electrical conductivity is enhanced. Additionally, it is possible to observe that the connection between grains appears poorer in the case of the film grown on FS, so that intergranular electron transport is expected to be harder than in the other two cases, which explains qualitatively the lower value of the electrical conductivity on FS.

Figure 4(b) shows the influence of substrates on Seebeck coefficient and plotted with respect to the inverse of temperature. The relation can be written as $S = (k/e)(E_s/kT + B)$; where E_s is thermopower activation energy and B is the heat of transport taken as temperature independent [28]. The value of E_s is calculated from the slope of linear fitting of the plots and belongs 0.04 to 0.05 meV. The values of E_σ are two order lower than the values of E_s which suggests that the conduction in these films are the result of thermally activated hopping mechanism [28, 30]. Due to Al³⁺ doping and oxygen vacancies, thin films show negative Seebeck coefficient on all substrates. Thin film AZO/FS shows higher value of Seebeck coefficient (-119/-203 μ V/K at 300/600 K) than thin films AZO/Al₂O₃ (-58/-126 μ V/K at 300/600 K) and AZO/STO (-90/-163 μ V/K at 300/600 K). In 2D materials and thin films, Seebeck coefficient is related to the phononic scattering by impurities, point defects and grain boundaries [31]. The higher roughness and poorer connection between grains in AZO/FS films respect to the films grown on single crystals may result in larger amount of phononic scatterers. This argument may be invoked to justify the larger Seebeck coefficient of AZO/FS in the whole range of temperatures.

From the values of Seebeck coefficient and electrical conductivity, we have calculated the power factor (PF) of thin films as $PF = \sigma.S^2$. Figure 5(a) shows the value of PF for thin films deposited on different substrates in the temperature range 300 K to 600 K. Thin films deposited on Al_2O_3 and fused silica shows higher value of power factor in whole range of temperature from 300K to 600 K than the thin film deposited on STO substrates. The value of PF is reported in table 1 at 300 K and 600 K. At 600 K thin film AZO/ Al_2O_3 shows slightly higher value of PF $0.43 \times 10^{-3} W^{-1}m^{-1}K^{-2}$ than thin films on other substrates.

Further, we measured thermal conductivity of thin films using time domain thermoreflectance technique and reported in table 1. We found the value of thermal conductivity is lower for thin film AZO/FS ($4.89 Wm^{-1}K^{-1}$) than thin films on single crystal substrates at 300 K. Which can be possible because of grain boundary scattering shown in figure 2 for thin film AZO/FS. And also TEM image figure 3 (i) shows randomly oriented layers which increase boundary scattering.

Dimensionless figure of merit $ZT = (\sigma.S^2).T/\kappa$ (where S: Seebeck coefficient; σ : electrical conductivity; κ : thermal conductivity; T: absolute temperature) [32] is calculated and reported in Table 1. The behavior of ZT values for thin films at elevated temperatures are estimated using out-of plane κ at 300 K (κ_{300K}) and shown in figure 5(b). This approach is used on the basis of the following facts: (i) in columnar films of ZnO whose morphology is quite similar to ours, in plane thermal conductivity values determined in different conditions are always higher than out-of plane values [33] and (ii) the thermal conductivity of ZnO films is found to decrease with increasing temperature [34]. Thin film AZO/FS shows the best ZT value 0.007 among all thin films at 300 K and predicted enhancement up to 0.046 at 600 K, 5-fold than the bulk of the same composition.

4. Conclusions

In this contribution, we proposed comparative study of the substrate effect on the growth mechanism of Al-doped ZnO (AZO) thin films for thermoelectric applications. The formation of the seed layer is the key that leads to the significant decrease of thermal conductivity ($4.89 \text{ Wm}^{-1}\text{K}^{-1}$ at 300K) and improvement of the thermoelectric figure of merit ($ZT = 0.046$ at 600K) of the AZO/fused silica (FS) thin films over the AZO/ Al_2O_3 and AZO/STO thin films and a bulk AZO. This result is encouraging for the practical applications of thermoelectric oxide thin films on cheap fused silica substrate. We are looking forward to the fabrication of tiny modules for ubiquitous harvesting of the waste heat generated by car engines, plants, incinerators at temperatures over 1000 K. Considering that the enhanced performance of AZO/FS film is due to the natural defects of the film (grain boundaries, point defects, etc.) there is still room for improvement with nano engineering approach, for example introducing artificial nanodefects for reducing thermal conductivity.

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Table caption:

Table 1: Electrical and thermal parameters for thin films and bulk pellet of AZO at 300K and/or 600 K.

Figure caption:

Fig1: XRD pattern (a): (θ - 2θ), (b) (ϕ -scan) of AZO thin film deposited on single crystal STO, Al_2O_3 and amorphous silica substrates at 400°C .

Fig2: SEM image of AZO thin film deposited on single crystal STO, Al_2O_3 and amorphous silica substrates at 400°C .

Fig3: First column shows cross-sectional TEM images with different magnification of AZO thin films deposited on STO (a), Al_2O_3 (d) and, FS (g) substrates; second column shows magnified TEM images of substrate and thin film interface deposited on STO (a), Al_2O_3 (d) and, FS (g) substrates; the third column shows magnified TEM images inside the film and the arrow shows the direction of c -axis deposited on STO (a), Al_2O_3 (d) and, FS (g: at the interface of thin film and the FS substrate) substrates, white dotted line shows random orientation of layers in thin film. Self-grown interface seed layer shown in (e) for AZO/ Al_2O_3 and (g) for AZO/FS.

Fig4: Electrical characterization:(a) $\log(\sigma T)$ vs $1000/T$ plots with solid lines of linear fit; thermoelectric characterization: (b) S vs $1000/T$ plots with solid lines of linear fit of AZO thin film deposited on single crystal STO, Al_2O_3 and amorphous silica substrates at 400°C .

Fig5: Thermoelectric performance: (a) power factor (PF) vs temperature; (b) dimensionless figure of merit ZT at elevated temperatures using thermal conductivity at 300 K for AZO thin film deposited on single crystal STO, Al_2O_3 and amorphous silica substrates at 400°C .

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Substrates	Grain Size	Electrical conductivity	Carrier concentration	Seebeck coefficient	Power Factor	Thermal conductivity	ZT
	nm (300 K)	σ (300 K/600K)	N (300 K)	S (300K/600 K)	PF (300K/600K)	κ (300 K)	(300K /600K)
		S/cm	10^{19} cm^{-3}	$\mu\text{V/K}$	$10^{-3} \text{ Wm}^{-1} \text{ K}^{-2}$	$\text{Wm}^{-1} \text{ K}^{-1}$	
STO	60	98/133	0.75	-90/-163	0.08/0.32	6.55	0.0036 /0.029
Al2O3	60	299/291	1.1	-58/-126	0.1/0.43	6.897	0.004/ 0.036
Fused Silica	90	74/93	0.8	-119/-203	0.11/0.37	4.89	0.007/ 0.046
AZO Bulk	--	206/152	--	-132/-150	0.35/0.34	34	0.0034 /0.014

Table 1: Electrical and thermal parameters for thin films and bulk pellet of AZO at 300K and/or 600 K.

Figures 1

Figures 2

Figures 3

Figures 4