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Transparent SnO-SnO₂ p-n Junction Diodes for Electronic and Sensing Applications

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The p-n junction is a simple but versatile structure used in many electronic devices such as diodes, junction transistors, solar cells and light-emitting diodes.^[1] Apart from the rectifying effect, p-n junction devices have been widely used in sensing applications, such as thermometer, photodetector and radiometer. Transparent all oxide based p-n junction diodes have been attracting increasing attention due to the encourging rectifying performance and the promise of achieving transparent circuits.^[2-5] As its name suggests, a p-n junction consists of serially connected p- and n-type semiconductors, where the operation of the device strongly depends on quality of the interface between n- and p-type semiconductor layers, which may be degraded by interfacial surface roughness, third phase impurity and the formation of interfacial layer. The interface quality is also related to the materials selection for n- or p-type transparent semiconducting oxides (TSOs). The oxide phase of metal with multi-valence state is more inclined to be oxidized or reduced when exposed to higher process temperature or extended processing time^[6, 7], which would lead to the formation of third phase impurity or interfacial layer, degrading the corresponding device performance. So far, a variety of n-type TSOs with decent electrical performance and visible-range transparency are available $[^{8}]$, including indium oxide (In₂O₃)^[9], zinc oxide (ZnO)^[4, 10], tin dioxide (SnO₂)^[11] and amorphous gallium-indium-zinc oxide (a-GIZO)^[3, 5, 12]. In comparison to n-type TSOs, the p-type

counterparts lag in performance. The most promising p-type oxide semiconductors include ternary Cu oxides^[13] [which include both Cu⁺ based delafossites (CuMO₂, M=AI, Ga, In, etc) and non-delafossites (CuSr₂O₂)], binary copper oxides (CuO and Cu₂O)^[5, 14, 15], spinel oxides (ZnM₂O₄, M=Rh, Co, Ir)^[3, 4, 16] and tin monoxide (SnO)^[17-20]. Among these, Cu₂O and SnO have been reported with higher p-type Hall mobility values due to the more dispersed valence band maximum, which results from the hybridization between the O 2p and Cu 3d (or Sn 5s) orbitals.^[15, 21-23] However, these two p-type oxides are known to be metastable phases, which has the tendency to be oxidized, forming stable phases with higher metal valence states.^[14, 20] The p-n junctions based on these two p-type oxide always suffer from the formation of defective interfacial layers. For example, about 80 nm interfacial layer was reported by Sathyamoorthy et al. in their p-SnO/n-SnO₂ junction diode, which exhibited an unexpectedly large ideality factor of 21.5, along with a small rectification ratio.^[24] If the interfaial layer thickness could be reduced or its quality could be improved, p-SnO/n-SnO₂ junctions with better perfromance can be realized.

In this report, we present p-SnO/n-SnO₂ junction diodes with ideality factor of \sim 3 and large rectification ratio (\sim 10³). The enhanced device operation, compared to previous studies on similar junction diodes, is likely related to a reduced interfacial layer thickness, which will be discussed later in the manuscript. In addition, a large temperature dependent knee voltage shift is observed which can be attributed to the large band gap of tin oxides and the existence of shallow states in p-SnO layer.

The n- and p-type conductivity of the SnO₂ and SnO layer was confirmed by Seebeck measurements, with Seebeck coefficient of -197 and +110 μ V K⁻¹ near room temperature, respectively. The Hall-effect measurement of p-type SnO thin film showed a hole mobility of 5.27 cm² V⁻¹ s⁻¹ and carrier concentration of 1.9×10¹⁷ cm⁻³. This mobility value is higher than the previous report by Y. Ogo et al.^[17] (2.4 cm² V⁻¹ s⁻¹) and E. Fortunato et al.^[18] (4.8 cm² V⁻¹ s⁻¹), the details about this high mobility thin film is shown in reference [20]. The density

functional theory simulations suggested that defects (Sn interstitial and oxygen vacancy) generated under Sn-rich growth conditions gave a more metallic character to the valence band of the SnO, which contributed a higher mobility.^[23] The p-type mobility of SnO thin films reported in recent 5 years are summarized in **Table S1**. Arrhenius plots of p-type SnO sample is shown in **Figure 1**, where the conduction behavior cannot be explained by a single thermal activation model but a phonon assisted thermally activated transport mechanism. Conductivity at higher temperature range (T>200 K) can be attributed to phonon assisted thermal activation while at the lower temperature range (T<200 K) is dominated by the phonon-assisted conduction mechanism.^[25] Our result is consistent with the previous report on transportation mechanism of SnO films by E. Fortunato et al.^[18] The calculated thermal activation energy is ~26 meV, which is smaller than the previous report^[18], indicating the existence of shallow states above the valence band. The more linear fitting achieved in conductivity vs 1/T^{1/4} curve (inset of **Figure 1**), agrees well with the Mott's theory, indicating a variable-range hopping or percolation conduction, which is consistent with E. Fortunato et al.^[18]

A schematic diagram for our p-n diode structure is shown in **Figure 2a**. The Ohmic contact between Au and SnO have been confirmed (**Figure S1**). **Figure 2b** shows the I-V characteristics of our p-n diode at various measurement temperatures, which clearly show rectifying behaviors. The I-V curve shifted to the negative direction with increasing measurement temperature. The knee voltage (V_{knee}) of each curve was determined by extrapolating the voltage-axis intercepts of the I-V curves, and the results are shown as inset to **Figure 2b**. The natural logarithmic I-V curves [ln(I_D) vs V] of the diode measured at different temperatures are shown in **Figure 2c**, in which an increasing operation current is observed at higher temperatures. The reverse saturation current (I_S) can be determined from the intercept of the linear part of the ln(I) vs V curve, and it is shown as inset to **Figure 2c**. It can be clearly seen that the I_S increases with rising temperature. According to the p-n diode operation equation^[1], I_D = I_S exp(eV (nkT)⁻¹-1), the ideality factor (*n*) of the diode can be

extracted from the slope of the $ln(I_D)$ -V curve (Figure 2c), and the results are shown in Figure 2d. The ideality factor values were determined to be \sim 3 and \sim 10, for the low voltage (0.5 V) and high voltage (2 V) regimes, respectively. The variable ideality factor is attributed to the influence of the serial resistance and the dominate operation mechanism, where tunneling at high voltage regime will lead to a higher ideality factor.^[26] The diode characteristic curve is plotted on logarithmic scale in Figure 2e, which shows the total reverse currents and was used to calculate the forward to reverse current ratio (I_f/I_r) . The I_f/I_r ratios are presented in Figure 2f which shows that the our SnO/SnO_2 p-n diode exhibits an I_f/I_r ratio of $\sim 10^3$ at $\pm 3V$. Temperature dependence of the device reverse saturation current is shown in Figure 2g. It is found that the reverse saturation current roughly follows the Mott's theory, i.e. 1/T^{1/4} dependence, indicating a variable range hopping conduction in a constant density of states near the Fermi level. Based on the band structure of SnO and SnO₂^[27], the energy band diagram for the p-SnO/n-SnO₂ junction is shown in **Figure 2h**. The energy barrier (ΔE_V) for hole injection was found to be 3.4 eV, which is close to our room-temperature measured V_{knee} value (3.27 V). The performance of our p-n junction diode is shown in Table 1, and is compared to previously reported similar p-n junctions using SnO as p-type oxide semiconudctor. It is clear that our device holds the largest I_f/I_r ratio (~10³) and a good ideality factor (~3.4).

Besides a better p-n junction operation, it should be noted that our p-n diode also shows a much higher V_{knee} drop rate of -20 mV °C⁻¹ (from 3.27 V at 25 °C to 2.15 V at 80 °C), which is quite favorable compared with silicon based diodes (-2 mV °C⁻¹)^[1]. We speculate that this phenomena comes from the large band gap (E_g) of tin oxides. The diode operation can be described by **Equation 1**^[1],

$$I_D = I_s \exp \frac{eV_D}{nkT} \tag{1}$$

where I_D is the forward current, I_S is the reverse saturation current, *e* is the electron charge, V_D is the applied voltage, *n* is the ideality factor, *k* is the Boltzmann constant and T is the ambient temperature on Kelvin scale. Then the forward voltage can be expressed as:

$$V_D = \frac{nkT}{e} \left(\ln I_D - \ln I_S \right) \tag{2}$$

It should be noted that I_D is not a function of T while I_S is. Then,

$$\frac{\partial V_D}{\partial T} = \frac{V_D}{T} - \frac{nkT}{e} \frac{\partial \ln I_s}{\partial T} = \frac{V_D}{T} - \frac{nkT}{eI_s} \frac{\partial I_s}{\partial T}$$
(3)

The reverse saturation current Is can be expressed as^[1]

$$I_{s} = eA\left[\frac{D_{P}}{L_{P}N_{D}} + \frac{D_{N}}{L_{N}N_{A}}\right]n_{i}^{2}$$

$$\tag{4}$$

with,

$$n_i^2 = BT^3 \exp\left(-\frac{eE_g}{kT}\right)$$
(5)

where A, B are constants. E_g is the band gap of the host semiconductor. Combining **Equations 4** and **5**, also considering D_P and D_N as a function of T^{-1} ,^[1] then, I_S can be expressed as follows,

$$I_{s} = CT^{2} \exp\left(-\frac{eE_{g}}{kT}\right)$$
(6)

where *C* is another constant. By taking **Equation 6** back into **Equation 3**, the relationship between forward voltage (V_D) and temperature (T) can be expressed as follows,

$$\frac{\partial V_D}{\partial T} = \frac{V_D}{T} - \frac{nE_g}{T} - \frac{2nk}{e}$$
(7)

Our p-n diode shows a V_{knee} of 3.27 V and $n\sim3$ at room temperature (T=298 K). The E_g can be taken as the average E_g value of SnO₂ (3.6 eV)^[8] and SnO (2.7 eV)^[17], which is ~3 eV. With these values, we calculated a $\partial V_D / \partial T$ slope of -19.7 mV °C⁻¹, which is close to our experimentally measured value (-20 mV °C⁻¹). The temperature dependent knee voltage shift

is normally related with the changing of p- and n-type carrier concentration in each layer, which is controlled by the relative position between the Fermi level and valence/conduction band. A large band gap will enlarge the change of carrier concentration in each type of semiconductor layer. Therefore, we attribute the large knee voltage shift to the large band gap of tin oxides. On the other hand, the Arrhenius plots of p-type SnO sample indicates a phonon assisted thermally activated mechanism. The E_a is quite small (26 meV) and can be treated as shallow states, which is more sensitive to temperature (much easier to be activated) compared with the deeper ones. We speculate this small E_a might also contribute the large temperature dependent knee voltage shift in our p-n diode, showing good potential for this diode in sensing applications.

Materials characterizations were also performed to understand the behavior of the reported p-n diode. X-ray diffraction (XRD) patterns of SnO and SnO₂ single layers and SnO/SnO₂ bilayer samples are shown in Figure 3a. The observed XRD peaks of the SnO film match well with the tetragonal α -SnO structure (JCPDS card No. 06-0395). Additional peaks from tetragonal metallic Sn (β -Sn, JCPDS card No. 04-0673) can also be found in the SnO diffraction pattern, which is consistent with our previous report on SnO thin films.^[20] In case of the SnO₂ film, XRD peaks corresponds to the (110), (101), (200) and (211) planes of tetragonal rutile SnO₂ were observed (JCPDS card No. 41-1445). The Scherrer equation was used to estimate the average grain size in the SnO and SnO₂ films, resulting with ~22.11 and ~10.05 nm, respectively. XRD patterns of the bilayer sample clearly show the peaks corresponding to the SnO and to SnO₂ phases. The peak at 50.5° may correspond to the (-1-41) plane of the intermediate triclinic phase Sn₃O₄ (JCPDS card No. 16-0737). Figure 3b shows the Raman spectra of the SnO₂ SnO₂ single layers and SnO/SnO₂ bilayer samples. In case of the SnO single layer sample, two peaks located at 109 and 210 cm⁻¹ can be observed, which are assigned to the B_{1g} and A_{1g} vibration modes of SnO, respectively.^[32] The Raman spectrum of the SnO₂ single layer sample shows peaks at 481, 631 cm⁻¹, corresponding to the

vibration modes of Eg and A1g, respectgively.^[33] In the bilayer film, the Raman spectrum is mainly coming from the SnO and SnO₂ phases. However, additional peaks at 129 and 152.5 cm⁻¹ are also detected, which may be assigned to vibration modes of the intermediate phase Sn₃O₄.^[33] The Raman results are consistent with the XRD analysis. The performance of the pn diode (i.e. ideality factor and I_f/I_r ratio) can probably be further improved if the amount of this thrid interfacial phase can be further reduced or eliminated. UV-visible transmission spectra of SnO, SnO₂ single layers and SnO/SnO₂ (SnO on top) bilayer samples deposited on glass substrates are presented in Figure 3c. The tauc plots of the SnO and SnO₂ films are shown in Figure 3d. The extracted optical band gaps are 2.68 and 4.1 eV, respectively, which match well with previous reports.^[8, 17] Figure 3e and f show the atomic force microscopy (AFM) images of the SnO₂ and SnO/SnO₂ (SnO on top) sample surfaces, respectively. The root-mean-square surface roughness of SnO₂ and SnO/SnO₂ are found to be 2.43 and 4.95 nm, respectively. Figure 3g and h give the statistical grain size results for both surfaces from the AFM data. The average grain size for the SnO/SnO₂ surface (~16.01 nm) is much larger than that of the SnO₂ surface (~6.70 nm), which is consistent with the grain size trend from the XRD patterns.

High resolution cross-sectional TEM (HRTEM) was performed to investigate the microstructure of the SnO/SnO₂ bilayer sample. The images are shown in **Figure 4**. The scanning TEM (STEM) micrograph in **Figure 4a** clearly shows the stack structure of the diode device [glass substrate/ITO(150 nm)/SnO₂(170 nm)/SnO(150 nm)]. The SnO₂ layer shows a columnar grain structure along the film growth direction, which was also observed in the SnO/SnO₂ bilayer sample reported by Sathyamoorthy et al.^[24] **Figure 4b and c** show selected area electron diffraction (SAED) patterns of the SnO and SnO₂ zones, respectively. The SAED pattern of SnO is taken from a grain for which the diffraction point matches well with the interplanar distance (*d*) of tetragonal α -SnO (JCPDS card No. 06-0395). For the SnO₂ case, concentric polycrystalline diffraction rings can be observed, with the diameters

matching well with the *d*-values of tetragonal SnO₂ (JCPDS card No. 41-1445). Figure 4d and e show the HRTEM micrographs of the SnO and SnO₂ zones, respectively. The microstructures inside the SnO and SnO₂ layers are quite different. In the case of the SnO film, large grains are observed; for the SnO₂ film, a polycrystalline columnar grain structure is observed. The grain size variation is consistent with the aforementioned XRD and AFM results. The different microstructures of the SnO and SnO₂ layers will lead to large stress in the interfacial zone. Figure 4f shows the HRTEM micrograph of the interfacial zone, in which ~30 nm interfacial layer can be observed. The microstructure inside this layer is different from both the SnO and SnO₂ layers. Fast Fourier transform (FFT) analysis was performed in selected areas enclosed by the dotted rectangles, and the results matched with the *d* value of the triclinic Sn_3O_4 phase (JCPDS card No. 16-0737), as depicted in Figure 4f. In fact, small crystallites of intermediate phase always exist to minimize the stress between the SnO and SnO₂ layers with totally different microstructures (as illustrated in Figure 4d, e and f). Further, since both SnO and SnO_2 are oxides of metal tin but with different oxygen coordination, considering the local disproportionate redistribution of internal oxygen, an interfacial layer with intermediate valence state phase (Sn_3O_4) is possible.^[20, 32] The presence of the intermediate phase is consistent with our XRD and Raman analysis. Compared with the previous literature report^[24] (~80 nm interfacial layer), the defective interfacial layer in our SnO/SnO₂ bilayer system is reduced (~30 nm), which may explain our improved device operation.

On the basis of the above mentioned material characterization results, our p-n diode can be seen to consist of n-type SnO_2 and p-type SnO layers with polycrystalline structures. Around 30 nm interfacial layer consisting of the intermediate phase of triclinic Sn_3O_4 with small crystallites is formed between the n-type SnO_2 and p-type SnO layers.

Although the ideality factor $(n\sim3)$ of our p-n junction is quite small compared with previous report $(n\sim21.5)^{[24]}$, it is still larger than the ideal value $(n\sim1 \text{ or } 2)$. This can be

explained by the defect-assisted tunneling mechanism.^[26, 34] Firstly, the thin Sn₃O₄ layer (~ 30nm) between p-SnO and n-SnO₂ could be regarded as a defective layer, offering large density of traps that require the carriers to go through by tunneling, which will increase the ideality factor of the junction away from 1 (or 2).^[26] Secondly, the mismatch between these two layers will also lead to a large ideality factor.^[35] From the TEM characterization, it is clear that large lattice mismatch exists between SnO and SnO₂, which may lead to large quantity of trap centers at the interface, contributing the unsatisfied ideality factor.

In summary, a p-n diode was fabricated by using p-type SnO and n-type SnO₂ semiconductors, sputtered from the same target. The SnO/SnO₂ based p-n diode shows an ideality factor of ~3 and I_f/I_r ratio ~10³. The deviation of ideality factor from ideal value was explained by the defect-assisted tunneling mechanism from the defective interfical layer. The p-n diode also exhibits a large temperature induced knee voltage shift of -20 mV °C⁻¹. The large knee voltage change with temperature is attributed to the large band gap of tin oxides and shallow states from the small activation energy in p-SnO layer. Our SnO/SnO₂ p-n diode has potential to be used as temperature sensing unit in transparent electronic applications.

Experimental Section

Device Fabrication: Commercial 150 nm indium tin oxide (ITO) coated glass was used as bottom electrode (n-contact) and substrate. The ITO layer was patterned by wet etching using a HCl: H₂O solution (75 vol%) at room temperature. An n-type SnO₂ layer (170 nm) was deposited by magnetron sputtering at a direct current (dc) power of 50 W, a deposition pressure of 4 mTorr and oxygen partial pressure (Opp) of 50%. The deposition was performed at room temperature. An ambient annealing in a tube furnace at 400 °C for 2 hr was applied to crystallize the as-deposited SnO₂ film. About 150 nm of p-type SnO was then sputtered on top of the annealed SnO₂ layer from the same target at a dc power of 20 W, deposition pressure of

1.8 mTorr and Opp of 9%. About 80 nm Au was e-beam evaporated as top contacts. Lift-off technique was used to pattern the SnO₂, SnO and Au layers. The whole device was then subjected to an ambient annealing at 200 °C for 1.5 hr in order to crystallize the p-type layer.

Device and Materials Characterizations: The Arrhenius plot was measured by Physical Properties Measurement System from Quantum Design, the resistance was measured by electrical transport option with AC frequency of 3.05 Hz. The temperature changing rate during measurement was 1.5 K min⁻¹. The current-voltage (I-V) characteristics of the p-n diode was measured by an Agilent B1500A semiconductor device analyzer in dark. Temperature dependent I-V measurements of the p-n diode were performed by an AirCool thermal stage SP72 from ERS electronic GmbH. The Seebeck coefficient was measured by the differential method using a commercial setup (RZ2001i, Ozawa Science Co Ltd., Nagoya, Japan). XRD patterns of the tin oxide films were obtained by a Bruker D8 discovery XRD system. Raman spectra were obtained at room temperature using a LabRAM ARAMIS Raman spectrometer (Horiba Scientific) with a 473 nm cobalt laser source excitation. AFM was performed by an Asylum Research (MFP-3D) scanning probe microscope in tapping mode. The AFM data was analyzed by the Gwyddion software. UV-visible transmittance spectra were obtained by an Evolution 600 UV-vis spectrophotometer (Thermo Scientific). HRTEM analysis of the bilayer sample was performed by a Titan ST TEM (FEI).

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Figure 1. Arrhenius plot of p-type SnO thin film, $1/T^{1/4}$ dependence is shown in the top right inset. Electrode configuration is shown in the bottom left inset.



Figure 2. (a) Schematic diagram of the p-n diode device. (b) Device characteristics at various temperatures, the V_{knee} values are illustrated as inset. (c) Diode operation curve on natural logarithmic scale with I_S value shown as inset. (d) Ideality factors in the low and high voltage ranges. (e) Diode operation curve on logarithmic scale and (f) $I_{fr}I_r$ ratios at various temperatures. (g) Temperature dependence of the device reverse saturation current. (h) Energy band diagram for the p-SnO/n-SnO₂ junction diode.



Figure 3. (a) XRD patterns and (b) Raman spectra of SnO, SnO_2 single layers and SnO/SnO_2 bilayer samples. The XRD reference peaks are shown as bars in the bottom. (c) UV-visible transmittance spectra of SnO, SnO_2 single layer and SnO/SnO_2 bilayer samples, (d) tauc plots for SnO and SnO_2 thin films. AFM images of the (e) SnO_2 and (f) SnO/SnO_2 surfaces. Statistics of the grain size on the (g) SnO_2 and (h) SnO/SnO_2 surfaces.



Figure 4. Cross-sectional TEM micrograph of the SnO/SnO_2 p-n diode device. (a) STEM micrograph of the device stack. SAED patterns of the (b) SnO and (c) SnO_2 samples. HRTEM micrographs of the (d) SnO, (e) SnO_2 single layer and (f) SnO/SnO_2 interfacial layer.

Materials		Method ^{a)}	T _{dep} ^{b)}	Substrate ^{c)}	μ_{Hall}	N _h	I _f ∕I _r	Range	n	V _{knee}	year	Ref
р	n		[°C]		[cm ² V ⁻¹ s ⁻¹]	[cm ⁻³]		[V]		[V]		
SnO	SnO:Sb	PLD	550	(001) YSZ	2.4	2.5×10 ¹⁷	-	±2.2	-	0.7	2011	[27]
SnO	ZnO	RFMS	RT	Glass	-	2×10 ¹⁸	12	±4.5	11.2	3	2013	[28]
SnO	SnO ₂	TE	300	Glass	-	-	-	±10	21.5	3.5	2014	[24]
SnO	SnO ₂	RFMS	100	Glass	0.13	7.3×10 ¹⁸	-	±7	-	2.3	2014	[29]
SnO	Si	EBE	RT	Si	-	1×10 ¹⁷	58	±2	5.5	1.1	2015	[30]
SnO	SnO ₂ :Sb	RFMS	200	(100) Quartz	3.34	2.3×10 ¹⁸	510	±6	6.4	2.9	2015	[31]
SnO	SnO ₂	DCMS	RT	Glass	5.27	1.9×10 ¹⁷	10 ³	±3	3.39	3.27	2015	This report

Explanation for column content and abbreviations in respective column. ^{a)}Method: preparation method for p-channel. (EBE: electron beam evaporation; PLD: pulsed laser deposition; RF/DCMS: radio frequency/direct current magnetron sputtering; TE: thermal evaporation.); ^{b)}T_{dep}: deposition temperature for p-type SnO. (RT: room temperature); ^{c)}Substrates (YSZ: yttria-stabilized zirconia); ^{d)}Range: applied voltage sweeping range.

The table of contents entry

Keyword: p-n junction diode, tin dioxide, tin monoxide, interfacial layer, temperature sensor

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Transparent SnO-SnO₂ p-n Junction Diodes for Electronic and Sensing Applications

ToC figure



The SnO/SnO₂ based p-n junction diode was fabricated by sputtering the semiconductors from the same target at room temperature. The p-n diode shows an ideality factor of ~ 3 and I_f/I_r ratio of ~10³. It also exhibits a large knee voltage shift with temperature of -20 mV °C⁻¹, which indicates that it can potentially be used in transparent temperature sensing applications.

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