

RECENT ADVANCES AND NOVEL APPROACHES OF P-TYPE DOPING OF ZINC OXIDE

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Abstract. Due to its high radiative stability and superior optoelectronic properties, such as wide direct band gap of ~3.4 eV and high exciton binding energy of ~60 meV, ZnO is considered for the fabrication of ultraviolet and visible light emitting and laser diodes, solar-blind photodetectors. Although proof-of-concept devices were already demonstrated, further progress in this area is slowed down by the difficulties of doping ZnO p-type. Here, we discuss problems associated with doping of ZnO, cover recent progress in this area, and discuss an alternative approach to increase p-type dopability via anion substitution, replacing oxygen with other group VI elements (S, Se, Te). We also propose that these anion-substituted alloys will be extremely promising for fabrication of photovoltaic devices, such as highly efficient thin film solar cells.

1. PROPERTIES AND GROWTH OF ZnO

There is significant technological and scientific interest in zinc oxide (ZnO) due to its unique physical and chemical properties [1]. It is radiatively hard wide band gap semiconductor ($E_g \sim 3.37$ eV) that can be easily doped n-type. Its band gap energy can be tuned by alloying with MgO and CdO from 7.9 eV to 2.3 eV [2], spanning from deep ultraviolet (UV) to visible regions of the spectrum. It has a higher exciton binding energy (E_b of ~60 meV) than other wide band gap semiconductors like GaN and SiC, leading to the efficient excitonic transitions at room temperature. In addition, owing to the availability of native substrates¹ and amenability to wet chemical etching, ZnO is an extremely promising material for the development of optoelectronic devices, such as

light emitting and laser diodes (LEDs and LDs) and solar-blind photo detectors, and transparent thin film transistors. ZnO nanostructures (nanoparticles, nanorods, nanobelts, etc.), which can be synthesized using inexpensive physical vapor transport techniques, were shown to be extremely promising for the application as gas sensors, due to the large surface to volume ratio [3], and microlasers due to the superior optical properties [4]. Furthermore, reports of high-temperature ferromagnetism in ZnO doped with transition metals (known as dilute magnetic oxide semiconductor) [5] coupled with nanosecond-long spin coherence time measured at low temperatures [6] makes this material extremely promising for future application in spin electronics (spintronics).² For comparison of ZnO with other wide band gap semiconductors see Table 1.

¹ Wafers with the diameter up to 4 inches are available from a number of companies including *Cermet Inc.*, *MTI Corporation*, and *Zn Technology Inc.*

² Use of quantum mechanical spin states associated with the charged particles to propagate information offers potential advantages over the use of charge alone, as in present-day technology. Spintronic devices, combining the advantages of magnetic metals and semiconductors, are expected to be non-volatile, versatile, fast, capable of simultaneous data storage and processing, and power efficient. They can be used in high-density data recording and storage, quantum computation, cryptography, and microelectronic.

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Table 1. Physical properties of wide band gap semiconductors.

Semiconductor	Crystal Structure	Band gap	E_g (eV) at ~ 6K	E_b (meV)
ZnO	Wurzite	Direct	3.44	63
ZnS	Zincblende	Direct	3.80	38
ZnSe	Zincblende	Direct	2.82	21
ZnTe	Zincblende	Direct	2.39	19
GaN	Wurzite	Direct	3.50	20
6H-SiC	Wurzite	Indirect	3.02	27

Table 2. Best parameters reported for ZnO films.

	$\Delta\omega$ <0002> peak	$\Delta\phi$ <10-11> peak	FWHM of band edge emission at 10K	electron density at room temperature	electron mobility at room temperature
Units	degree	degree	meV	cm ⁻³	cm ² V ⁻¹ s ⁻¹
Parameter	>0.01	>0.02	1.5 meV	5 x 10 ¹⁵	300
Growth	PLD	PLD	MBE	PLD	PLD
Technique					
Substrate	ScAlMgO ₄	ScAlMgO ₄	GaN	ScAlMgO ₄	ScAlMgO ₄
Reference	[27,81]	[27,81]	[82]	[81]	[27]

High crystalline quality ZnO films are successfully deposited using a number of growth platforms, including molecular beam epitaxy (MBE), chemical vapor deposition (CVD), pulsed laser deposition (PLD), and reactive sputtering. Elemental sources (Zn and ozone or oxygen plasma), ceramic targets (ZnO), and metalorganic precursors (dimethylzinc and diethylzinc) are used with equal success. The best parameters reported for the ZnO films are shown in Table 2. Some of these growth techniques, such as PLD, can be used to deposit films and device structures only on the small wafers, up to 1 inch in diameter, and are more applicable for academic research. Others, such as sputtering, are easily scalable and allow straightforward integration with modern microelectronic industry.

2. P-TYPE DOPING OF ZnO

However, further progress in this area is stalled by the difficulties of doping ZnO p-type. It is generally acknowledged that high enough doping levels are difficult to achieve both due to the background n-type doping originating from the presence of H impurities [7], O vacancies (V_O) [8], and Zn interstitials (Zn_i) [9], and due to the large acceptor activation energies and / or low solubility of commonly used group-V (N, P, As, Sb) and group I (Li) dopants. In

addition, a slow transition from p-type to n-type conductivity was observed by a number of groups. It was tentatively assigned either to the acceptor migration from the substitutional to the interstitial position and / or to the hydrogen diffusion [10,11].

In spite of these difficulties, there are reports of ZnO doped p-type with group V (N [12,13] P [14,15], As [16-20], and Sb [21,22]), and group I (Li [23]) elements. Co-doping with two potential acceptors (N and As) [24] or acceptor and donor (N and Al) [25,26] was also studied. In the case of the acceptor-donor co-doping, the improvement is believed to be primarily due to the higher solubility of the forming N-Al-N complex. A temperature modulation epitaxy technique was also applied to achieve N-doped p-type ZnO [27]. Nitrogen-doped layers were grown at low-temperature (400 °C) to increase solubility followed by the deposition of thin undoped layers at high temperature (950 °C) to improve crystalline quality. In addition, doping with other elements, such as Ag and Bi, was explored [28,29]. These results are summarized in Table 3.

Unfortunately, most of these results were extremely challenging to reproduce and nontrivial to interpret. For example, ZnO films grown by PLD on GaAs substrates were reported to be p-type. This was explained by the As diffusion from the GaAs substrate into the ZnO layer [16]. However, addi-

Table 3. Summary of attempts to grow p-type doped ZnO films.

Dopant	Growth Technique	N_A (cm ⁻³)	μ (cm ² / V s)	P (Ohm cm)	Reference
N	CVD	1.06x10 ¹⁸	0.34	17.3	[12]
N	CVD		ZnO:N remained n-type		[13]
P	Diffusion		ZnO:P layer was p-type		[14]
P	Sputtering	1.9x10 ¹⁶ - 3.8x10 ¹⁹	6.9 - 40.1	0.02 - 8.1	[15]
As	PLD	10 ¹⁸ - 10 ²¹	0.1 - 50	10 ⁻⁵ - 10	[16]
As	Sputtering	9x10 ¹⁹	4	-	[17]
As	PLD	2.4x10 ¹⁷ - 1.1x10 ¹⁸	-	2.2 - 6.7	[18]
As	CVD	1.45x10 ¹⁸	1.92	2.3	[19]
As	PLD	4x10 ¹⁹	2	0.05	[20]
Sb	MBE	1x10 ¹⁶	10	6	[21]
Sb	PLD	1.9x10 ¹⁷	7.7	4.2	[22]
Li	PLD	6.04x10 ¹⁷	1.75	5.9	[23]
N + As	CVD		Mixed conductivity		[24]
N + Al	Sputtering	2.52x10 ¹⁷	0.43	57.3	[25]
N + Al	Sputtering	1.45x10 ¹⁸	1.62	2.64	[26]
N	PLD		8		[27]
Ag	PLD	4.9x10 ¹⁶ - 6.0x10 ¹⁷	0.29 - 2.32	34 - 54	[28]
Bi	MBE		ZnO:Bi remained n-type		[29]

tional effects, such as formation of an interfacial Zn₂As₂O₇ layer [30], Ga diffusion into the ZnO film [31,32], and Zn diffusion into the GaAs substrate [33] were reported by other groups. In addition, isolated As atoms should act either as deep acceptors (As is incorporated substitutionally at the O position - As_O) and donors (As is incorporated substitutionally at the Zn position - As_{Zn}) or remain amphoteric (As is incorporated interstitially - As_i). Thus, p-type conductivity was explained by the formation of a complex with two spontaneous Zn vacancies (As_{Zn}-2V_{Zn}) [34]. Clearly, data interpretation was not straightforward and the origin of the p-type response remained controversial.

3. ZnO – BASED DEVICES

Due to the difficulties with achieving highly p-type doped ZnO, proof of concept devices that did not employ p-type ZnO layer, such as n-ZnO/p-SrCu₂O₂ [35], n-ZnO/p-SiC [36], n-ZnO/p-Si [37], ZnO-based metal-insulator-semiconductor [38], n-GaN/ZnO nanowire/p-GaN [39], and n-ZnO/p-GaN were fabricated [40,41]. The last approach was widely explored due to the low lattice mismatch between ZnO and GaN and possibility of band gap engineering using Al_xGa_{1-x}N alloy. Thus, p-Al_xGa_{1-x}N/ZnO/n-Mg_xZn_{1-x}O LEDs operating at high temperatures, up

to 650K, were demonstrated [42]. In addition, there were a few recent reports on the fabrication of homojunction LEDs, indicating continuous progress in this area [43-47]. ZnO-based LEDs produced using spray pyrolysis were particularly promising [48]. Possibility to use such a low-cost and easily scalable technique justifies further development of this material system. Results of these research efforts are summarized in Table 4.

In addition, ZnO-based UV photo detectors - another important area of ZnO application - were demonstrated by a number of research groups [49-51]. These devices are promising for the fabrication of the early warning missile defense systems, can be used for high temperature flame detection, for the monitoring of ozone layer, and in solar astronomy.

However, homojunction LEDs suffered from high turn-on-voltage, reaching 12 V in the case of Be_xZn_{1-x}O-based LED, and strong deep level defect-related emission. While high turn-on-voltage can be explained by the significant resistivity of the p-type layer, deep level emission may arise due to the inferior crystalline quality of the p-type layer. Although there are reports that a large dopant concentration can be incorporated into ZnO without degradation of a crystalline quality [52,53], a poorer in-plane and out-of-plane alignment, larger tilt and twist, is often observed [54]. This may be due both to the defects

Table 4. Characteristics of ZnO-based LEDs.

n-Layer	p-layer	λ (nm)	Turn-on voltage (V)	Reference
ZnO	SrCu ₂ O ₂	382	3	[35]
ZnO:Al	4H-SiC	385	3.8	[36]
ZnO	Si	385	Under reverse bias	[37]
ZnO		380, 550	8.9	[38]
GaN:Si	GaN:Mg	382, 430	5	[39]
ZnO:Ga	GaN:Mg	430	3	[40]
ZnO:Ga	GaN:Mg	375	3.3	[41]
Mg _x Zn _{1-x} O	Al _y Ga _{1-y} N	390	3.2	[42]
ZnO	ZnO:N	Operate only below 200K		[43]
Be _x Zn _{1-x} O:Ga	Be _x Zn _{1-x} O:As	363, 388, 500	12	[44]
ZnO	ZnO:N	423, 523	3.7	[45]
ZnO	ZnO:As	388, 500	4	[46]
ZnO	ZnO:N	430	7	[47]
Mg _x Zn _{1-x} O	Mg _x Zn _{1-x} O:N+In	450, 525	3.4	[48]

introduced by the dopant, such as acceptor taking interstitial position (P_i , As_i , Sb_i) paired with the formation of oxygen vacancy (V_O), as well as to the lower growth temperatures used to increase solubility.

Therefore, a novel approach is clearly needed to achieve high crystalline quality p-type ZnO layers necessary for the fabrication of low turn-on-voltage LEDs, LDs, and p-n junction photo detectors.

4. GROWTH AND DOPING OF ANION SUBSTITUTED ZnO

It was recently suggested that p-type dopability of ZnO can be improved through the anion substitution, replacing oxygen with other group-VI elements, such as S, Se, and Te. The band alignment between ZnO and zinc chalcogenides (ZnS, ZnSe, ZnTe) is plotted in Fig. 1 [55]. Due to the much higher valence band of zinc chalcogenides, the acceptor activation energy is lower in the anion-substituted alloys ($ZnO_{1-y}X_y$, $X = S, Se, Te$), making them much more amendable to p-type doping.

While the cation substitution is widely used both for the band-gap engineering ($Be_xZn_{1-x}O$, $Mg_xZn_{1-x}O$, and $Cd_xZn_{1-x}O$) [56-58] and for the magnetic doping ($Mn_xZn_{1-x}O$ and $Co_xZn_{1-x}O$) [59,60], anion substitution is much less studied, with the polycrystalline films being mostly investigated [61,62]. This is particularly surprising since this approach was previously employed to overcome problem of bipolar doping in $ZnSe_xTe_{1-x}$, where high p-type and n-type levels were reported for Te-rich and Se-rich alloys, respectively [63,64]. A significant success was

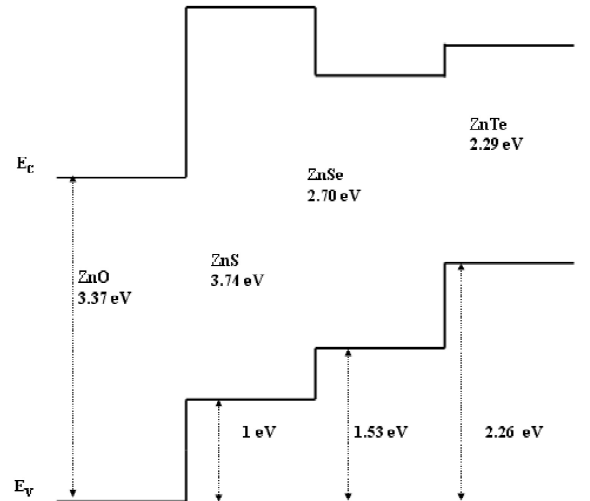


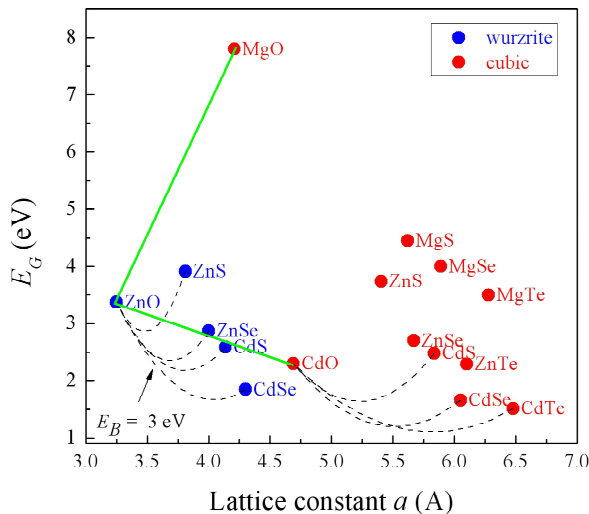
Fig. 1. Calculated [55] band alignment between ZnO and zinc chalcogenides (ZnX , $X = S, Se, and Te$).

achieved with the so-called d-doped films where p-type dopant (N) was introduced into Te-rich nanoislands embedded within the undoped ZnSe matrix. The net acceptor concentration ($[N_A - N_D]$) $\sim 6 \times 10^{18} \text{ cm}^{-3}$ was achieved in the samples containing less than 3% of Te ($ZnSe_{0.97}Te_{0.03}$) [65]. This is an order of magnitude higher than that achieved by any other technique.

There is a report on the epitaxial growth of oxide-rich $ZnO_{1-y}S_y$ ($y < 0.15$) using PLD [66]; on the Se-rich side up to 1.35% of oxygen were doped in ZnO_ySe_{1-y} [67,68]. A much larger oxygen content ($x < 10\%$) was incorporated into the $ZnO_xS_ySe_{1-x-y}$

Table 5. Summary of attempts to grow anion substituted ZnO alloy.

Alloy	Composition (%)	Growth Technique	Crystalline Quality	Bowing Parameter(eV)	Reference
$\text{ZnO}_{1-y}\text{S}_y$	$0 < y < 100$	Sputtering	Polycrystalline film	3	[61]
$\text{ZnO}_{1-y}\text{S}_y$	$0 < y < 100$	Atomic Layer Deposition	Polycrystalline film	3.6	[62]
$\text{ZnO}_{1-y}\text{S}_y$	$0 < y < 15$	PLD	Epitaxial film	—	[66]
$\text{ZnO}_y\text{Se}_{1-y}$	$0 < y < 1.35$	MBE	Epitaxial film	—	[67]
$\text{ZnO}_y\text{Se}_{1-y}$	$0 < y < 1.3$	MBE	Epitaxial film	8	[68]
$\text{ZnO}_x\text{S}_y\text{Se}_{1-x-y}$	$0 < x < 10$	MBE	Epitaxial film	5 - ZnOS 9 - ZnOSe	[69]
$\text{ZnO}_{1-y}\text{S}_y$	$0 < y < 5$ $96 < y < 100$	Chemical Vapor Transport	Bulk crystal	3	[70]

**Fig. 2.** Band gap energy plotted vs. lattice constant for selected II-VI semiconductors. Green solid lines and black dotted lines show the estimated band gaps for $\text{Me}_x\text{Zn}_{1-x}\text{O}$ and $\text{ZnO}_y\text{X}_{1-y}$ alloys, respectively.

films closely lattice matched to GaP substrates. There, epitaxial stabilization prevented phase separation [69]. In addition, bulk $\text{ZnO}_{1-y}\text{S}_y$ ($y < 0.05$ and $y > 0.96$) crystals were grown using chemical vapor transport technique [70]. All these results, summarized in Table 5, indicated a significant band gap bowing.

The valance band of $\text{ZnO}_{0.81}\text{S}_{0.19}$ is estimated to be 410 meV higher than that of ZnO. In this case, the acceptor activation energy decreases from 220

meV to 5 meV for a heavily nitrogen doped sample [62]. With N becoming a shallow acceptor, high p-type doping levels are much easier to achieve.

5. ANION SUBSTITUTED ZnO FOR OPTOELECTRONIC AND PHOTOVOLTAIC DEVICES

It is important to note that the band gap energy of $\text{ZnO}_{0.81}\text{S}_{0.19}$ remains above 3 eV. Thus, an efficient blue emission is expected from the $\text{ZnO}_y\text{S}_{1-y}$ -based LEDs. In addition, an enhanced photoconductivity was observed in ZnO films co-doped with Te and nitrogen [71]. Therefore, an enhanced performance of the UV photo detectors based on the anion substituted ZnO is also expected.

With the cation substitution, band gap of the $\text{Me}_x\text{Zn}_{1-x}\text{O}$ (Me = Be, Mg, Cd) can be tuned from the deep UV to the visible range of spectrum. In particular, a 2.6 eV emission line (475 nm) was observed from the $\text{Zn}_{0.84}\text{Cd}_{0.16}\text{O}$ films, indicating that pure green $\text{ZnO}/\text{Cd}_x\text{Zn}_{1-x}\text{O}$ LEDs can be fabricated [72]. With the anion substitution, band gap energy of the $\text{Me}_x\text{Zn}_{1-x}\text{O}_y\text{X}_{1-y}$ can be further adjusted throughout the whole visible range (band gap of $\text{Zn}_x\text{Cd}_{1-x}\text{O}_y\text{Se}_{1-y}$ can be tuned from 3.4 to 1.7 eV) down to the near-IR region of the spectrum, (band gap of $\text{CdO}_x\text{Te}_{1-x}$ can decrease to ~1.1 eV assuming bowing parameter of ~3 eV). We plot band gap energies versus lattice parameters for the selected II-VI semiconductors in Fig. 2.

Thus, this material is extremely promising for the fabrication of full color and pure white LEDs,

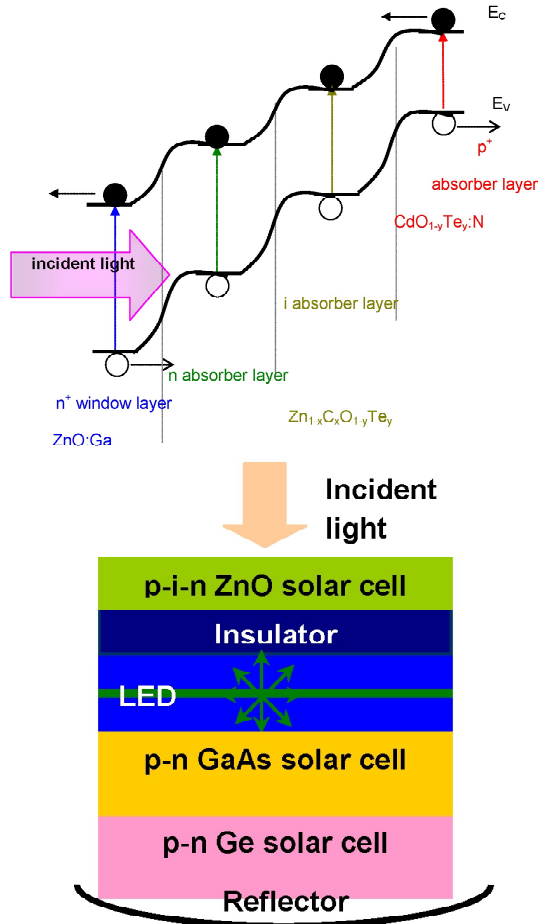


Fig. 3. The schematics of a $\text{Zn}_{1-x}\text{Cd}_x\text{O}_{1-y}\text{Te}_y$ -based multi-layer graded band gap solar cell consisting of 4 layers with decreasing energy gaps (n^+ -window layer with $E_g \sim 3.4$ eV, n -absorber layer, i -absorber layer, and a p -type absorber layer with $E_g \sim 1.5$ eV).

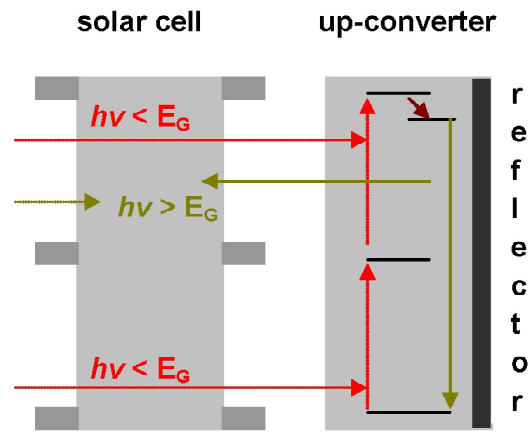


Fig. 4. The schematics of a system based on a wide band gap $\text{Me}_x\text{Zn}_{1-x}\text{O}_y\text{X}_{1-y}$ -based solar cell and an up-converter electronically isolated from each other. Up-converter consists of a tandem of two narrow band gap solar cells, such as Ge and GaAs, driving a $\text{Me}_x\text{Zn}_{1-x}\text{O}_y\text{X}_{1-y}$ -based LED. The sub-band-gap light transmitted by the wide band gap solar cell ($h\nu < E_g$) is partially up-converted into high-energy photons ($h\nu > E_g$), which are subsequently absorbed by the wide band gap solar cell. A reflector is located behind the up-converter to maximize collection [79,80].

where red-green-blue emission is obtained from the heterostructures that are grown on the same substrate and differ only in the composition of the quantum well region [73], and photovoltaic devices, in particular, for the design of the multi-layer thin film solar cells [74-76]. In these cells, a wide band gap ZnO:Ga ($E_g \sim 3.4$ eV) can be used as a transparent top n -type contact, CdTe:N can be employed as a bottom p -type contact ($E_g \sim 1.5$ eV), and the layers of intermediate composition can be utilized as the absorber layers, as shown in Fig. 3. Due to the compositional gradient, multi-layer solar cells will possess the gradual reduction of the band gap and the variation of electrical conduction type from n^+ to p^+ from the front to the back. The number of layers can be increased to any number in order to absorb a major part of the solar spectrum and to produce

smooth slopes of energy band edges, improving the photo-generated charge carrier separation and collection. A significant improvement in efficiency is expected for these devices since two major mechanisms responsible for the efficiency loss – lattice thermalization of high-energy photons and transmission of low-energy photons – are minimized. In addition, since ZnO is already considered as a transparent top contact layer in $\text{Cu}(\text{In}_x\text{Ga}_{1-x})\text{Se}_2$ -based solar cells [77,78], such a substitution will simplify device fabrication process.

Moreover, $\text{Me}_x\text{Zn}_{1-x}\text{O}_{1-y}\text{X}_y$ -based LEDs can be epitaxially integrated with the $\text{Me}_x\text{Zn}_{1-x}\text{O}_{1-y}\text{X}_y$ -based solar cells on the top of narrow-band gap solar cells, such as Ge/GaAs multitandem structures. In this case, two narrow band gap solar cells, absorbing low energy photons, will drive LED emitting high-

energy photons. These high-energy photons will be sequentially absorbed by the electrically isolated $\text{Me}_x\text{Zn}_{1-x}\text{O}_{1-y}\text{X}_y$ -based wide band gap solar cell in addition to the photons received directly from sun, as shown in Fig. 4. A significant improvement in the efficiency predicted for such a system [79,80] makes it extremely promising for the space applications where high efficiency is of primary importance.

4. CONCLUSION

In conclusion, we reviewed recent progress in the growth of ZnO-based materials and fabrication of ZnO-based devices. We covered difficulties of doping ZnO p-type, which is known as a major obstacle for the fabrication of low turn-on-voltage light emitting and laser diodes and p-n junction photo detectors. We presented and discussed novel and extremely promising approach to achieve highly p-type doped layers via anion substitution, replacing oxygen with other group-VI elements (S, Se, and Te). We also suggested that anion substituted alloys are of particular interest for the use in the photovoltaic devices, such as highly efficient thin film solar cells.

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