Rectifying ZnO–Na/ZnO–Al aerogels p-n homojunctions

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ABSTRACT

Semiconductor ZnO aerogels were synthesized by a sol–gel process with different concentrations (2.5–7.5 wt.%) of Al (n-type) or Na (p-type) and dried under supercritical CO₂. The materials were calcined at 500 °C to remove the organic content and to crystallize the ZnO. The microstructure of the ZnO-based aerogels comprises a porous structure with hexagonal and platelet-shaped interconnected particles. The bandgap of the aerogels doped with Al decreased significantly compared to pure, undoped ZnO aerogels, while their specific surface area increased. For the electrical characterization of the ZnO–Na/ZnO–Al junctions, the doped ZnO aerogels were deposited on commercial glass substrates coated with indium tin oxide (ITO) by drop casting method. The I–V curves of the p–n homojunctions revealed a characteristic diode rectifying behavior.

1 Introduction

Zinc oxide (ZnO) is a semiconductor with hexagonal wurtzite structure, direct wide band gap (~ 3.37 eV) with low toxicity, low processing cost, large exciton binding energy (60 meV) at room temperature and high chemical stability, which has attracted substantial research attention due its potential application in electronics and optoelectronics [1–3]. The ZnO can form various types of nanostructures that have been largely applied in photodetectors [4], piezoelectric devices [5], photocatalysis [6], gas sensors [7, 8], supercapacitors [9], light-emitting diodes (LEDs) [10, 11], solar cells [12], Schottky diodes [13] and p–n junction diodes [14–16].

As a result of intrinsic defects such as oxygen vacancies and Zn interstitials [17], ZnO shows a natural thermally stable n-type conduction behavior that can be enhanced by dopants, such as Al [18], Sb [19], Ga and Sn [20]. The p-type conduction, on the other hand, is hindered mainly due to the self-compensating effect and low solubility of acceptor dopants, hence, one of the biggest challenges in making p–n ZnO homojunctions is to achieve successful p-type doping [16, 21]. Some studies obtained n-type ZnO using K [22], Na [23], P [24] and N [25] as dopants. As a way to mitigate these effects and obtain p-type ZnO, Na have been suggested as dopant, since theoretically it can increase its solubility in ZnO [23, 26], which results in a stable p-type conductivity

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and allow the manufacturing of ZnO p-n homojunctions.

A variety of different shaped nanostructures, such as nanorods, nanotubes, nanoneedles, nanowires, nanoflowers, nanospheres [27], plates [28], tetrapods [29], among others, can be deposited in different types of substrates to obtain ZnO films and layered junctions. Many deposition techniques can be used to develop ZnO thin films, including pulsed laser deposition [30], spray pyrolysis [7, 31], sol-gel [32, 33], RF sputtering [34] and chemical vapor deposition [35]. Among the deposition techniques, the sol-gel process stands out as a bottom-up approach for the production of electronic devices due to its controllable reaction conditions and low process temperatures, that allows the incorporation of organic and inorganic materials [36].

The simplicity of the sol-gel method followed by drying under critical conditions using a supercritical fluid, such as CO₂, allow the production of porous materials with high porosity, low density and high surface area called aerogels [18, 37]. The aerogel structure, i.e., the high specific surface area and porous structure form interconnected solid networks that allow electronic conduction [38] and can improve the effective contact by forming a large contact surface to form the interface (junction) between films.

Therefore, this work reports the production and characterization of Na-doped ZnO (p-type) and Al-doped ZnO (n-type) aerogels, synthesized by the sol-gel process with different concentrations. The I-V characteristics of the p-n junctions the measurements were carried out for the individual metal oxides layers and for the p-n homojunctions fabricated by depositing the Na-doped ZnO onto an Al-doped ZnO layer through a simple method (drop casting).

2 Materials and methods

2.1 Reagents

All the following reagents in this work were used as received: Zinc chloride (Lafan), absolute ethanol (99.8%, Neon), polyacrylic acid (Sigma Aldrich), deionized water, propylene oxide (99%, Aldrich), sodium hydroxide (Vetec), aluminum chloride hexahydrate (Sigma Aldrich), diethylene glycol (Vetec),

acetone (P.A., Neon), isopropanol (99.8%, Neon) and silver conductive paint (Eletrolube).

2.2 Synthesis of the aerogels

The ZnO aerogels were obtained through an epoxy/sol-gel template route, based on the route proposed by Chen et al. [39]. 1.0 g of polyacrylic acid dissolved in distilled water was added under stirring to a 2 M ZnCl₂ ethanolic solution. Then, 0.07 mol of propylene oxide was added to gel the solution, and it was poured into molds until gel formation. After obtaining the gels, they were aged in an ethanol bath for 7 days. Finally, the gels were submitted to supercritical CO₂ drying (at 31 °C and 1072 psi) in a Tousimis Autosamdri 931 dryer, to form the ZnO aerogels. For ZnO aerogels doping with Na and Al, concentrations of 2.5, 5.0 and 7.5 wt.% of sodium hydroxide or aluminum chloride were used, by adding them into the ethanolic solution of ZnCl₂, and following the same sol-gel and supercritical drying procedure described above. Finally, the Na and Al doped ZnO and ZnO aerogels were calcined in air at 500 °C at a heating rate of 10 °C/min for two hours to obtain crystalline ZnO.

2.3 Device manufacturing

To manufacture the devices, first a suspension of aerogels was obtained, starting from 0.05 g of aerogel dispersed in 2.5 mL of diethylene glycol. This suspension was then deposited using the drop-casting method on glass substrates and on ITO coated glass substrates, previously washed in acetone and isopropanol ultrasonic bath, and heated at 120 °C/30 min to form thin films. This coating-drying cycle was 5 times repeated, as shown in Fig. 1. For the formation of the junctions, layers of ZnO-%Na, ZnO-%Al and ZnO-%Na/ZnO-%Al were deposited, and the metallic contacts were made with a silver conductive ink and an Al wire to carry out the electrical measurements.

2.4 Characterization

The structural analyzes of the aerogels were carried out by transmission electron microscopy (TEM, JEOL-JEM 1011), at a maximum acceleration voltage of 100 kV. The crystalline and compositional analysis was evaluated by X-ray diffraction (XRD, Phillis

Fig. 1 Schematic diagram of the device fabrication

X-Pert) with $\text{CuK}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$), scanning at 2θ with an interval between 5° and 100° in $5^\circ/\text{min}$ steps. The specific surface area of the aerogels was obtained using the Brunauer, Emmett and Teller (BET) technique (Quantachrome NOVA 2200e) by nitrogen adsorption at 77 K, with degas of 100°C for 1 h to remove moisture. The Tauc plot method was used to obtain the optical band gaps of aerogels through UV–Vis spectrophotometer (Biospectro SP-220) in a scanning range of 320–900 nm. To analyze the surfaces of thin films of aerogels deposited on ITO coated glass substrates, they were sputtered with gold and analyzed by scanning electron microscope (JEOL JSM-6390LV). To evaluate the electrical properties of the devices, conductivity measurements were performed using the collinear four-point probe method on the aerogel films deposited on glass. By deposition on ITO coated glass, three sets of devices were prepared: ZnO-%Na, ZnO-%Al and ZnO-%Na/ZnO-%Al. The Al and Na doped films on ITO coated glass and the ZnO p–n homojunctions were analyzed through their current–voltage characteristic curves using an SMU (Agilent, B2912A).

3 Results and discussion

Figure 2 shows the XRD spectra of the ZnO-Na[%] (Fig. 2a) and ZnO-Al[%] (Fig. 2b) samples after heat treatment at 500°C . In both diffractograms it is possible to see a high crystallinity with well-defined

Fig. 2 XRD spectra of ZnO aerogels with different dopant concentrations: **a** ZnO-Na and **b** ZnO-Al

peaks that correspond to the hexagonal wurtzite crystal structure of ZnO (JCPDS 36-1451), showing that the structure of ZnO, as well as its crystallinity, is not affected by the incorporation of Na and Al dopants. Additionally, there is no presence of peaks corresponding to metallic Al or Na or any other oxide phases, which suggests that Al and Na ions were incorporated into the ZnO lattice or that the amount used in the doping is below the detection limit of the equipment. No significant peak shift in 2θ are observed for both Na and Al doped ZnO (insets in Fig. 2a and b), except for the slight shift to lower angles displayed by ZnO doped with the highest concentration of Na (7.5 wt.%), due to substitution of Zn^{2+} (ionic radius $r = 0.74 \text{ \AA}$) by Na^+ with larger ionic radius ($r = 0.95 \text{ \AA}$). The crystallinity of this sample is also reduced, characterized by the broadening and intensity decrease in the XRD peak. In

literature, broader and less intense ZnO diffraction peaks with the doping of Al was reported [40, 41]. However, ZnO samples doped with Al change in crystallinity could be detected (Fig. 2b).

Figure 3 shows TEM micrographs of the ZnO-based aerogels after thermal treatment at 500 °C. The inset in Fig. 3a displays a photograph of an as-prepared undoped ZnO aerogel, which exhibits a characteristic opaque white color.

The TEM images of the pure ZnO (Fig. 3a and b) and ZnO-Na[5%] aerogels (Figs. 3c and d) reveal agglomerate structures composed of irregularly shaped particles of different sizes. In the ZnO-Al[5%] sample, Fig. 3e and f, a structure formed by agglomerated irregular particles and rod-shaped structures can be observed. A typical aerogel structure is shown in Fig. 3e, where a contiguous solid

network comprised of interconnected sub-micrometer sized particles, surround pores with similar sizes. After heat treatment an increase in crystallinity is expected; however, this is also accompanied by an increase in particle size and loss of surface area, which is due to the tendency of adjacent particles to coalesce, forming larger particles [39, 42, 43].

Table 1 summarizes the BET specific surface area and the electrical conductivity of the ZnO aerogels. Untreated pure ZnO aerogel exhibited a specific surface area of 152 m²/g. After heat treatment an expected loss of surface area was observed [2, 44]. This evident surface area drop is also justified because the samples experience self-combustion during heat treatment. The doped aerogels exhibit, on the other hand, higher surface areas, when compared to pure ZnO aerogels, which may be due to

Fig. 3 Structure characterization of aerogels by: **a** ZnO aerogel image before thermal treatment and TEM micrographs of the aerogels post thermal treatment: **b** and **c** ZnO, **d** and **e** ZnO-Na[5%], **f** and **g** ZnO-Al[5%]. Inset in Fig. 3a is a photograph of an undoped ZnO aerogel

charged nanoparticles present in the pores of the aerogel. The Al-doped ZnO aerogels have higher surface areas than the Na-doped aerogels. However, as the concentration of dopants increases, the surface area follows opposite trends for both Al and Na-doped aerogels, probably a result of an excessive number of reducers, which is likely to result in an excessive supply of carbon residues, causing a decrease in the surface area.

The measured conductivity values are presents in Table 1 for the calcined samples of Na-doped, with conductivities in the order of 10^{-7} to 10^{-5} S/cm. The conductivity values obtained for the Al-doped ZnO samples are in the order of 10^{-8} S/cm. High resistivity values in the order of $10^8 \Omega \text{ cm}$ were also reported for Al-doped ZnO films [45]. No increase in conductivity was observed with the increase in the percentage of Al doping. In some articles [46, 47], the conductivity values increase with doping compared to pure ZnO.

The optical band gap obtained by the Tauc plot method are shown in Fig. 4 for the ZnO-Na[%] aerogels (Fig. 4a) and ZnO-Al[%] aerogels (Fig. 4b).

The estimated optical band gap value of the pure ZnO aerogel is ~ 2.53 eV, which is lower than the theoretical band gap of crystalline ZnO (3.37 eV), and may be related to the presence of vacancies and other intrinsic impurities [48]. Doping with Na and Al in ZnO aerogels led to a decrease in their optical band gap values, which is a result of lattice modifications by Al and Na ions. It is also interesting to note that doping with Al is much more effective, with band gap values remaining below the value of undoped ZnO aerogels, while for Na, only the lowest

Fig. 4 Tauc plots and band gap values for ZnO aerogels with different concentrations of: **a** ZnO-Na[%] and **b** ZnO-Al[%]

concentration returned lower band gap, indicating that there is a limit for the doping to be efficient in ZnO.

The SEM micrographs of the pure and doped ZnO samples deposited on ITO-coated glass substrates are shown in Fig. 5. In the samples with pure and doped ZnO (Figs. 5a-c) it is possible to notice clusters of structures with different shapes, as already observed by TEM (Fig. 3). The calcined ZnO aerogels are composed of platelet-shaped and hexagonal particles with sizes ranging from hundreds of nanometers to few micrometers. It can be seen from Fig. 5d that the deposited material is irregularly distributed over the glass substrate, as a result of the drop casting method that does not allow for control and uniformity of deposition.

Figure 6 shows the I-V curves for pure, undoped and Na and Al-doped ZnO aerogels deposited on ITO-coated glass substrates. The inset

Table 1 Specific surface area (S_{BET}) and electrical conductivity of the Na and Al doped ZnO and ZnO samples, after thermal treatment at 500 °C

Material	S_{BET} [m^2/g]	Conductivity [S/cm]
ZnO	< 1	1.6×10^{-5}
ZnO-Na[2.5%]	23.0	1.1×10^{-7}
ZnO-Na[5%]	1.2	3.7×10^{-5}
ZnO-Na[7.5%]	7.0	8.0×10^{-7}
ZnO-Al[2.5%]	43.3	4.4×10^{-8}
ZnO-Al[5%]	52.3	6.4×10^{-8}
ZnO-Al[7.5%]	24.9	6.1×10^{-8}

Fig. 5 SEM micrographs of the aerogels suspensions deposited on the ITO-coated glass substrate: **a** ZnO, **b** ZnO-Al[5%], **c** ZnO-Na[5%] and **d** cross-section of ZnO-Na[5%]

figures illustrate the schematic of the measurement performed. For all measurements, the same settings and polarity of the applied potential were maintained for the contacts. The I-V curve of ZnO/ITO (Fig. 6a) presents an almost linear behavior, which can be attributed to a small potential barrier formed between ZnO and ITO [35]. With the junction of ZnO-Na[2.5%]/ITO (Fig. 6b) this potential barrier increases, but this linear behavior of the curve is still present. However, with an increase of Na doping to 5% it is possible to notice a rectifying behavior, typical of junction diodes. With the doping of 7.5% Na this rectifying behavior is attenuated. Similar to the curves of pure ZnO and Na-doped junctions, the ZnO-Al[2.5%]/ITO curve (Fig. 6c) presents an almost linear characteristic, and with 5% doping of Al the formation of the potential barrier at the junction results in the non-linearity of the curve. The I-V curve of ZnO-Al[7.5%]/ITO junction displays a rectification behavior, but with a symmetry inversion compared to the ZnO-Na[2.5%]/ITO junction, which indicates

that conduction of ZnO-aerogel based semiconductors is resulted from different charge carriers, i.e. electrons and holes, for Al-doped ZnO (n) and Na-doped ZnO (p), respectively. Figure 6d shows the I-V measurements of the junction between the Al or Na doped ZnO aerogels and the Ag electrodes. All samples exhibit a linear behavior, confirming that the ohmic contact was established between the electrodes and the semiconductors.

Figure 7 shows the schematic of the assembled devices and a view of the band diagram of the constructed junctions, along with their I-V curves. The I-V curves of all three symmetrically assembled ZnO-Na[2.5%]/ZnO-Al[2.5%]/ITO, ZnO-Na[5%]/ZnO-Al[5%]/ITO and ZnO-Na[7.5%]/ZnO-Al[7.5%]/ITO junctions exhibited a rectifying behavior.

The electrical behavior of the ZnO-based p-n devices was evaluated using the I-V curves to obtain the key electrical parameters summarized in Table 2.

For the junction doped with 2.5%, 5% and 7.5% turn-on voltages of 0.66 V, 0.96 V and 0.84 V were

Fig. 6 I-V curves of **a** ZnO/ITO, **b** ZnO-Na[%], **c** ZnO-Al[%] and **d** I-V dependencies of the silver contact on n-ZnO and p-ZnO. The insets show the device configuration and the I-V plot in semilog form

determined, respectively. The rectification ratio was calculated by the ratio between the current in forward and reverse bias (I_F/I_R), for the same absolute value of applied voltage. The rectification ratios of the ZnO doped with 2.5%, 5% and 7.5% were, respectively, 60.9, 4.0 and 6.2 at ± 1.5 V. From the logarithmic plot, the reverse saturation current (I_0) was obtained by extrapolating the curve in the linear region intercepting the y-axis. The smaller reverse saturation current, closer is the junction to an ideal diode [49].

Considering the thermionic emission model, the current (I) in the region of forward bias is given by Eqs. (1) and (2):

$$I = I_0 \left[\exp\left(\frac{qV}{nk_B T}\right) - 1 \right], \quad (1)$$

$$I_0 = AA^* T^2 \exp\left(-\frac{q\phi_B}{k_B T}\right), \quad (2)$$

where k_B is the Boltzmann constant, T is the absolute temperature, q is the electronic charge, ϕ_B the barrier height, A is the effective area and A^* is Richardson constant. The ideality factor (n) and the barrier height (ϕ_B) were obtained from the slope of the curve $\ln I$ - V . The lowest calculated ideality factor is 10.8 for the 2.5% of dopants device. For the junctions doped with 5% and 7.5%, the calculated ideality factors are significantly higher. The ideality factors values found for the junctions are similar, or higher, in the case of 7.5% doping, to the ideality factors reported in the literature, between 4.3 and 25 for ZnO p-n junctions [14–16].

Table 3 shows a comparison of results obtained from the I-V characteristics of the fabricated ZnO p-n homojunction with those observed for ZnO p-n junctions reported in the literature. The rectification ratio obtained for the ZnO-Na/ZnO-Al device with 5 wt% of dopants is 60.96 at ± 1.5 V, which is similar

Fig. 7 **a** The simplified energy band diagram and junction schematic. I-V characteristics of **b** ZnO-Na[2.5%]/ZnO-Al[2.5%]/ITO, **c** ZnO-Na[5%]/ZnO-Al[5%]/ITO and **d** ZnO-Na[7.5%]/ZnO-Al[7.5%]/ITO. The insets show the I-V plot in semilog form

Table 2 Summary of the parameters obtained by the I-V curves of the produced devices

Devices	I_F/I_R	V_{th} [V]	I_0 [A]	n	ϕ_B [eV]
ZnO-Na[2.5%]/ZnO-Al[2.5%]	60.96	0.66	7.01×10^{-5}	10.87	0.60
ZnO-Na[5%]/ZnO-Al[5%]	4.00	0.96	4.26×10^{-4}	24.34	0.55
ZnO-Na[7.5%]/ZnO-Al[7.5%]	6.28	0.84	9.27×10^{-4}	72.53	0.53

Table 3 Comparison of results of electrical measurements with other studies in the literature

Junction	I_F/I_R	n	Reference
ZnO-Na/ZnO-Al (2.5 wt.%)	61 at ± 1.5 V	10.9	This work
ZnO-Bi/ZnO	57 at ± 5 V	4.3	Singh et al. [50]
ZnO-Al,N/ZnO	20 at ± 2 V	3.8	Chiu et al. [51]
ZnO-Na,N/ZnO-Eu	12.5 at ± 5 V	7.6	Swapna et al. [52]
ZnO-Al,N/ZnO-Al	90 at ± 2 V	4.3	Lu et al. [16]
Pd/ZnO-Cu/ZnO	996 at ± 5 V	3.7	Agarwal et al. [53]
ZnO/ZnO-AlN	13.4 at ± 4 V	11.8	Balakrishnan et al. [54]
ZnO-As/ZnO	—	3–25	Ryu et al. [15]

or higher than those observed for ZnO p-n junctions reported by most studies. However, the values of ideality factors found for the junctions are similar or

superior, in the case of 7.5 wt% of doping, to the ideality factors reported in the literature. In summary, this comparison of the data reveals that the

devices developed in this work work are comparable with those reported in the literature.

The high ideality factors, deviating from an ideal p–n junction ($n=1-2$), suggests that there are multiple charge transport mechanisms that make the experimental values obtained for the manufactured device diverge from the thermionic emission model. Therefore, further studies are needed to improve the quality of the p–n junctions [16, 55].

4 Conclusions

Pure ZnO aerogels and doped with Na or Al (2.5, 5 and 7.5 wt%) were obtained by sol–gel process using the epoxy/template methodology with zinc chloride as precursor and dried under CO₂ supercritical conditions. After heat treatment at 500 °C the ZnO-based aerogels crystallized in hexagonal wurtzite phase of zinc oxide. The microstructure Devices were fabricated with n-type bottom layer of ZnO-Al[%] and a p-type top layer of ZnO-Na[%] deposited onto the ITO-coated glass substrate. The I–V curves for the developed devices showed non-linear behavior with rectification characteristics as expected for a p–n junction. From the curves, key parameters associated with diodes were also determined, such as the ideality factor, barrier height and rectification ratio. The highest calculated rectification ratio calculated was 60.9 for the ZnO-Na[2.5%]/ZnO-Al[2.5%] device.

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Authors contribution

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Karla N. Mukai, Joseane C. Bernardes and Daliana Müller. The first draft of the manuscript was written by Karla N. Mukai and all authors commented on previous versions of the manuscript. Carlos R. Rambo acquired funding and

supervised the work. All authors read and approved the final manuscript.

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Data availability

All data used to support the findings of this study are included within the article.

Declarations

Conflict of interest The authors declare that there is no conflict of interest regarding the publication of this paper.

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