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Chemisorbed CO_2 molecules on ZnO nanowires (100 nm) surface leading towards enhanced piezoelectric voltage

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ABSRACT

Here, we have studied the behavior of ZnO based piezoelectric mechanical transducer in the presence of CO_2. We have analyzed reduction-oxidation mechanism of CO_2 gas on ZnO nanowire surface and studied its behavior for the enhancement piezoelectric output voltage. Rise in piezoelectric potential for various CO_2 exposures time intervals from 30 min to 3 h has been observed. Chemiresistive behavior of CO_2 modified ZnO nanowires, is being explained by the generation of high piezoelectric voltage. ZnO nanowires based nanogenerator has been exposed to 30 min, 60 min, 90 min, 120 min and 3 h in a sealed chamber at 20 ppm, corresponding output piezoelectric voltages of 503.9 mV, 1.15 mV, 1.559 V and 1.795 V have been recorded respectively by using Picoscope 5204. It has also been observed that output voltage value has been saturated after 2 h of CO_2 exposure. ZnO nanowire based VING (vertically integrated nanowire generator) structure is being used for the study under minute external force of ~ 100 nN. Growth orientation of ZnO nanowires has been ensured along c-axis perpendicular to substrate.

1. Introduction

ZnO is promising group II–VI semiconductor material having bandgap of 3.37 eV. Its wide range of applications in piezoelectric, optoelectronic devices, gas sensing applications, bio-sensors and in self powered nano systems makes it a versatile material [1–6]. Nanopiezotronics has emerged as hot area of research now days, in which both piezoelectric and semiconducting properties are coupled to generate rectifying currents and piezoelectric potentials. Numerous mechanical transducers are being reported which used tiny external forces of few nN to produce piezoelectric output voltage pulses. Technique of harvesting mechanical energies from the ambient sources has led us to develop self powered nanoscale systems [7–10].

At nanoscale, ZnO nanowires/nanorods have shown more superior results as compared to other nanostructures like Si nanowires and carbon nanotubes. Properties like high aspect ratio, crystalline structure, high density and well aligned growth orientation makes them more prominent [11–16]. ZnO nanowires/rods have been extensively used for gas sensing purposes because of their Chemiresistive nature toward different gases like NO_2, CO, CO_2, NH_3, $H_2SC_2H_5OH$ [17–28] and First self powered ZnO based gas sensor is reported by Z L Wang [29] ZnO nanowires are intrinsically *n*-type material that is why chemically and electrically quite active. Typically for *n*-type materials, oxidizing agents like Oxygen captures the vacant sites and trap free electrons and increase the resistance of the material. Likewise for *p*-type materials, reducing agents donate the electrons to increase the conductivity of the material [30–33]. REDOX (reduction-oxidization) behavior of ZnO nanowires has been used to enhance the piezoelectric potential of energy harvester. ZnO nanowires based nanogenerator has been exposed to CO_2 for various time intervals from 30 min to 3 h approximately and a gradual rise in the output piezoelectric potential has been observed. Large surface to volume ratio of ZnO nanowires have provided more vacant sites. These vacant sites have been modified by chemisorbed species. Oxygen has the tendency to oxidize ZnO oxide surface quickly at

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room temperature, however at elevated temperature 150 °C > it showed desorption tendency as well. Oxidizing gases tend to enhance the depletion layer and so as the internal resistance of ZnO nanowires. CO_2 molecules adsorbing on ZnO nanowire surface react with already adsorbed O^{-} and extract more electrons from the conduction band of ZnO nanowires. Conduction band electrons decrease and the resistance of ZnO nanowires tends to increase. By reducing free charge carrier concentration from ZnO nanowires, reverse leakage current has been reduced and consequently enhancing piezoelectric potential is achieved. Maximum output voltage of 1.795 V with a maximum power density 215.8 mW/cm^2 for an exposure time for 2 h has been achieved. it has been also observed that further exposure has not produced any significant rise in output voltage which assures us that all vacant sites have been captured by chemisorbed CO_2 molecules.

2. Materials and methods

We have purchased all reagents of analytical grade (98%) from Sigma Aldrich and used them without further purification. ITO (indium tin oxide) coated PET (poly ethylene terephthalate) are first cleaned ultrasonically by deionised water and acetone for 10 min each respectively then dried in the air. 0.1 M solution of Zinc acetate dihydrate $(CH3COO)_2{\cdot}2H_2O]$ is used to grow seed layer on the substrates, afterwards PET substrates are annealed for 30 min at 50 °C. We prepared equimolar solution (1:1) of HMTA $[C_6H_{12}N_4]$ and Zinc nitrate hexahydrate $[Zn\ (NO_3)_2.6H_2O]$ to grow ZnO nanowires on PET substrates. Aspect ratio of ZnO nanowires is being dependant on growth time in the nutrient solution, similarly density of the nanowires was found dependant on the concentration of nutrient solution [5,6]. Procedural route of the growth of ZnO nanowires is shown in Fig. 1.

PET substrates are used as bottom electrode, ZnO nanowires as the intermediate part and gold sputtered electrode is upper electrode to construct VING (vertically integrated nanowire generator). VING has been exposed to CO_2 gas at 20 ppm in locally made gas chamber for different time duration ranging from 30 min to 3 h piezoelectric voltage has been measured after every 30min. exposure of CO_2. Picoscope 5204 is being connected to VING after each exposure to measure voltage changes in the output voltage graphs. Minute external force ~100 nN is applied to produce bending in nanowires for the generation of piezoelectric potential. ZnO being intrinsically *n*-type material and CO_2 as oxidizing gas has increased the internal resistance of the nanowires and enhanced the output voltage.

3. Results and discussions

1- ESEM Philips XL30 (resolution 4 nm) has been used to study the surface morphology of ZnO nanowires. SEM results of 100 nm ZnO nanowires grown on ITO coated PET substrates are shown in Fig. 2 (a, b, c, d). Highly dense structure of ZnO nanowires is clear from Fig. 2 (a, b). Vertical growth and dense structure both were necessary for VING. Dense structure ensures the maximum number of nanowires will contribute in high output power generation. Magnified image of ZnO nanowires as shown in Fig. 2 (c, d) indicated narrow spacing near the top edges of nanowires. It was quite essential because during an external stress nanowires need some space to bend. The production of piezoelectric potential was due to the bending of nanowires. It's a quasi array of ZnO nanowires of 100 nm. The average diameter of nanowires is around 100 nm as indicated my SEM images. However, we have also calculated the individual diameter of nanowire by using image analysis software imageJ [34]. In first step, SEM image is copied in the software and converted the image to 8-bit image then afterwards scale was calibrated as imageJ does not know about SEM image scale. Second step is binarization of gray scale image, by the using the contrast feature of the software only top area of nanowires is selected and rest of the potion is deleted. In third step the radius of selected nanowire is determined by using the relation

$$A = \pi r^2 \tag{1}$$

Then radius "r" is multiplied by 2 to get the diameter of individual nanowire. Histogram represents the diameter distribution of ZnO nanowires which is exactly in agreement with SEM Images. SEM results are closely in agreement with XRD pattern shown in Fig. 3. XRD pattern has been obtained by SHIMADZU-6000 X-ray diffractrometer using source Cu Kα (λ = 1.54° A). XRD peaks were matched by the card (JCPDS Card no 36–1451). Dominating sharp peak along (002) direction verified vertical growth of nanowires from PET substrates. There are few other weak peaks along (100), (102) and (110) planes but are not significant. Fig. 4 is the schematic diagram of mechanical nanogenerator and ZnO nanowire modified by CO2 molecules.

Intrinsically, ZnO wurtzite structure exhibit piezoelectric phenomenon under external applied force. In an unstrained condition, cations (Zn^{2+}) and anions (O^{2-}) are stacked over one over the other and their charge centre coincide with each other. As the external force is applied to crystal the charge centre disturbs and cations and anions move apart that results an electric dipole in the crystal. Cations tend to accumulate at one end of the crystal and consequently anions to the other end of the crystal and piezoelectric field start to develop within the crystal. Piezoelectric potential develops between two ends of the crystal, which is the basis of piezoelectric nanogenerator [9]. In VING, the deliverance of piezoelectric potential to the external stages is possible only if one end of structure has schottky contact at the one end of crystal is essential. It ensures the flow of electron only in one direction i.e. through the external circuit. It works like barrier to the electrons and prevents the flow of electron through (leakage current) the nanowire. For an ideal schottky contact the electron affinity of semiconductor must be less than the work function of metal. It has been already discussed in our earlier reported work [6,7].

Fig. 5 (a) ensures the schottky contact in between ZnO nanowires and

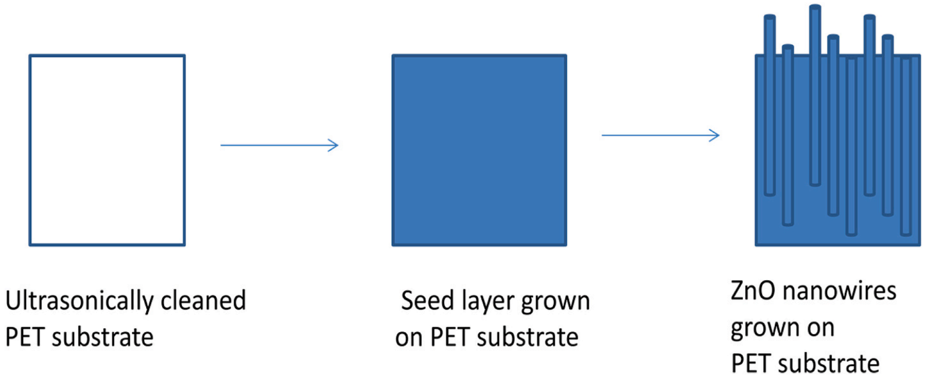


Fig. 1. Schematic diagram of ZnO nanowires growth.

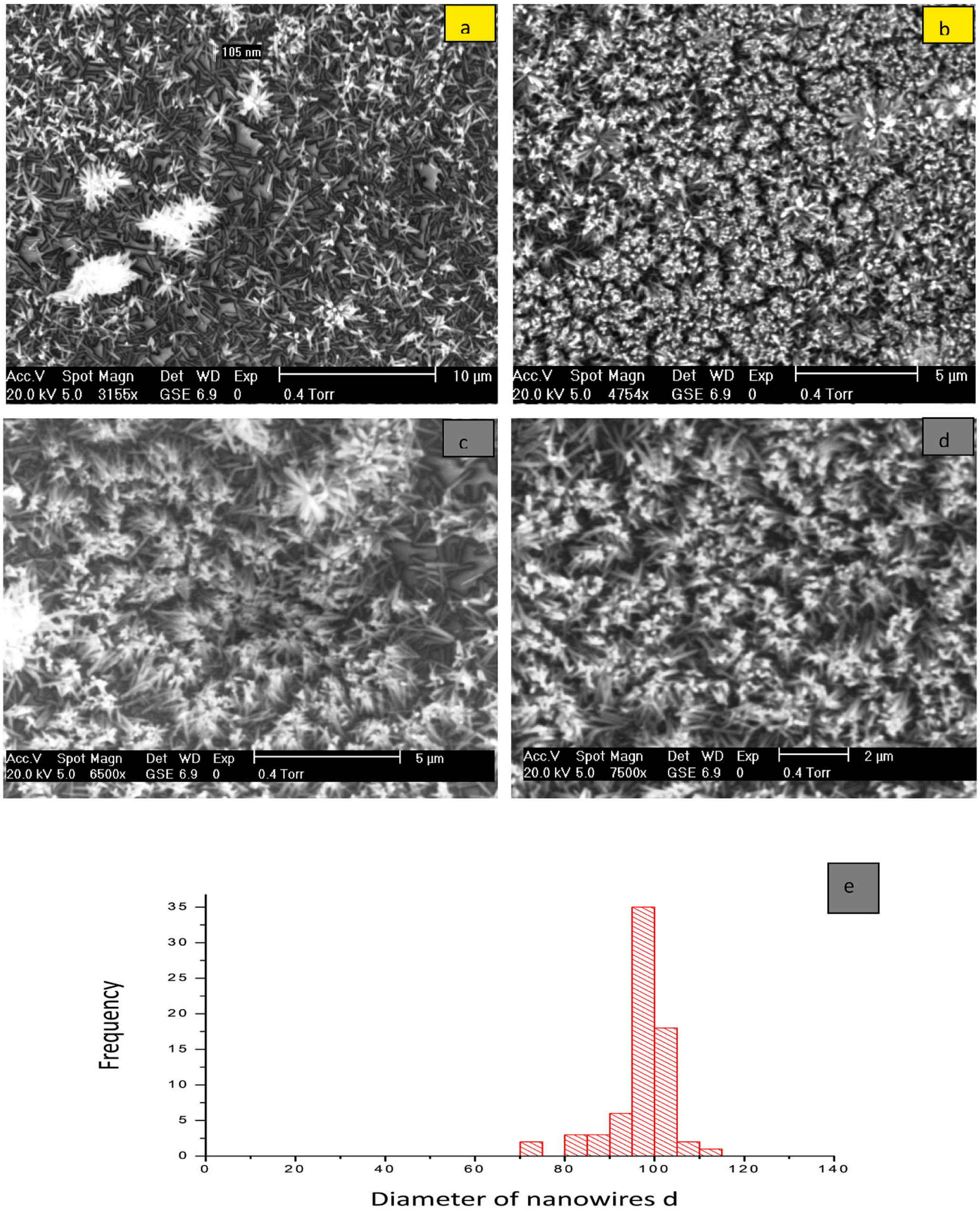


Fig. 2. (a, b, c, d) SEM image of 100 nm ZnO nanowires grown on PET substrate, at low and High magnification respectively(e) histogram showing diameter range of ZnO nanowires.

upper electrode. Periodic voltage peaks indicating production of piezoelectric potential generated by ZnO nanowires which is being detected by picoscope 5204. Schottky contact at least at one end of nanogenerator is essential. It ensures all the piezoelectric potential generated by nanowires must be delivered to the external circuit. It allows the electron to flow in only one direction only i.e. through the external circuit and prevents reverse leakage current through the nanowires.

In VING, minute external force is applied through the light plastic roller. As it has discussed earlier it will generate piezoelectric potential within nanowire and electrons tend to pile up on the top end of the nanowire giving rise to the conduction band Fermi level at the top relative to the bottom end of the nanowire due to which electrons flow from top to bottom electrode via external circuit and positive voltage peak is detected by the picoscope. As electrons will accumulate at the bottom end they will consequently rise up the conduction band and Fermi level at the bottom and an equilibrium state is achieved but as the external force is removed piezoelectric potential in nanowire perishes and to achieve balanced charged condition all the accumulated electrons at the bottom will flow back to top electrode via external circuit and opposite voltage peak is being detected by the picoscope.

To ensure the stability of the device, we have tested forward and reverse cycles of VING ten times. We have examined quite carefully that obtained piezoelectric output voltage values are true voltage generated values from the VING or not and it is depicted by the histogram in Fig. 5 (b). It is quite clear that occurrence of positive voltage and negative voltage peaks is exactly in agreement to the voltage value in the graph shown in Fig. 5 (a). Same procedure has been carried out after each voltage measurement to check the accuracy of VING.

VING has been exposed to CO_2 atmosphere at 20 ppm in sealed chamber for 30 min we have observed net rise of 125 mV in output voltage. It verifies the occupancy of gas molecules to some of the

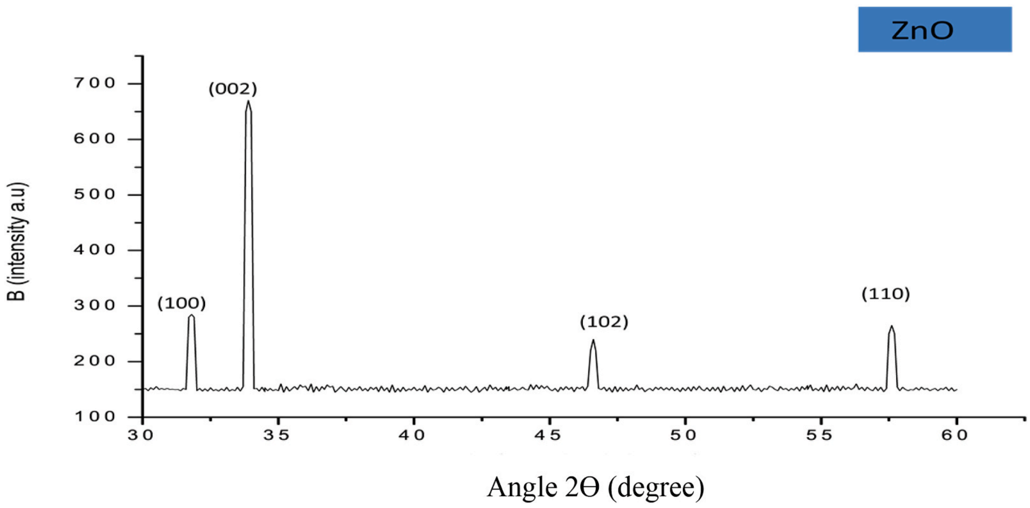


Fig. 3. XRD pattern of vertically grown ZnO nanowires.

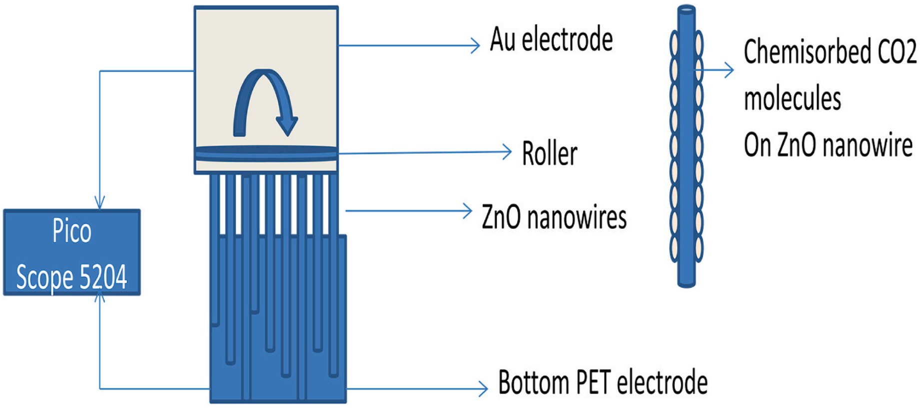


Fig. 4. Schematic diagram ZnO based energy harvester and ZnO nanowire modified with CO_2 molecules.

intrinsically present defects on ZnO nanowire. The adsorption of the gas molecules on ZnO nanowires could be understood in two steps, first O adsorption and in second step CO_2 adsorption. Oxygen is second largest gas present in the atmosphere. Chemisorptions phenomenon of CO_2 molecules on ZnO nanowire surface has produced Edge effects and reduced electron concentration. Due to edge effects the internal resistance of ZnO nanowires is increased which is highly desired. Due to high electron negativity (3.44) of oxygen, it plays a crucial role in adsorption process on ZnO nanowire surface. Intrinsically, ZnO nanowires are *n-type* nanostructures which have number of surface defect sites. Oxygen due to loan pair of electrons finds an easy way to get adsorbed on the ZnO nanowire surface and changed into ion (O_2^-). It forms an ionic layer on the surface of ZnO nanowires which helps other gas molecules to get adsorbed on the surface. Thus layer works as receptor function. Although diatomic oxygen is non-reactive in gas state but while interacting with ZnO nanowire surface, it works as an electron acceptor agent. It adsorbs excess electrons available on ZnO nanowires (*n-type*) surface and gets ionized [16,33–36].

$$O_2 + e^- \rightarrow O_2^- \tag{2}$$

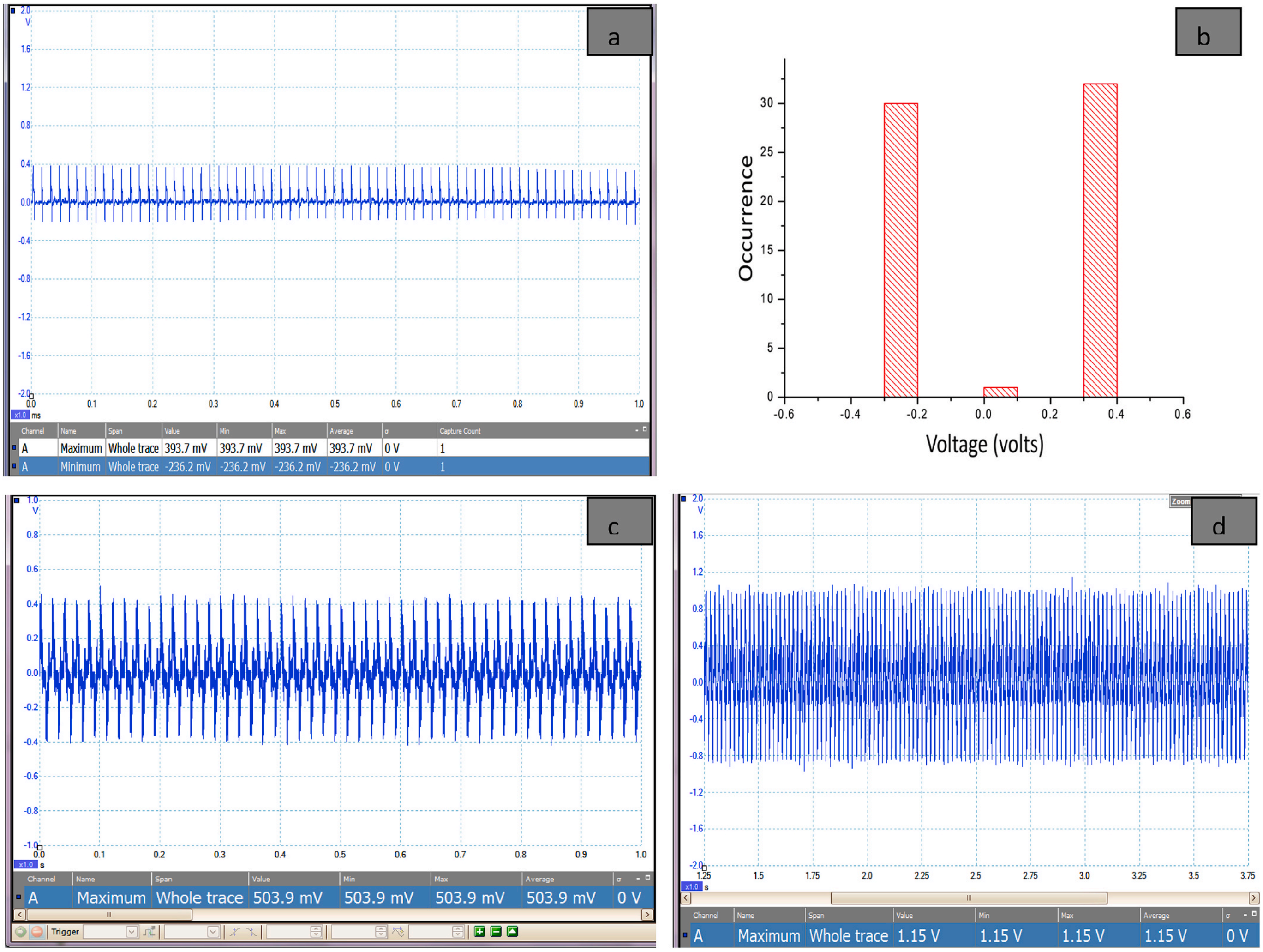
$$CO_2 + e^- \rightarrow CO_2^- \tag{3}$$

$$CO_2^- + O^- + 2e \rightarrow CO + 2O^{2-} \tag{4}$$

The adsorption phenomenon is being explained by the above reactions. In which, first step is the adsorption of O on surface defect sites of ZnO nanowires and in second step CO_2 adsorbs the free electrons and turns into CO_2^- and in third step CO_2^- reacts with adsorbed oxygen molecules and captures two more free electrons. During the process it is evident that population of free electrons is being further reduced and the internal resistance of the nanowires is increased. The reduction of free electrons from ZnO nanowire surface is highly favorable for ZnO based VING. These free electron cause reverse leakage current through the nanowires which act as bad factor. For high output piezoelectric nanogenerators, reverse leakage current through the nanowires must be minimized. Pure ZnO nanowires are intrinsically *n-type* material and exhibit large aspect ratio giving rise to number of electrons in structure which is termed as surface defect [37]. It has been clearly observed in the output voltage graphs that there is gradual rise in output voltage with the increase in exposure time of CO_2. Rise in output voltage has been observed until all excess electrons are being absorbed by first by O_2 and then by CO_2 and after that voltage value has been saturated as shown in Fig. 5(g). It has been observed that further exposure of CO_2 has not given rise in piezoelectric output voltage.

Humid condition could have negative impact on its receptor function but dry atmosphere is highly desirable for such type of phenomenon. As the electrons are being trapped by the oxygen, it creates depletion width for the electrons to surpass which consequently reduces the flow of the current through the ZnO nanowires and internal resistance of the nanowires increases [38].

Chemisorbed of CO_2 molecules have also triggered a depolarization process within ZnO nanowires. it induced an opposite electric field inside nanowires which depleted the piezoelectric potential inside nanowires. Depolarization process has reduced electronic output of ZnO



Channel	Name	Span	Value	Min	Max	Average	σ	Capture Count
A	Maximum	Whole trace	393.7 mV	393.7 mV	393.7 mV	393.7 mV	0 V	1
A	Minimum	Whole trace	-236.2 mV	-236.2 mV	-236.2 mV	-236.2 mV	0 V	1

Channel	Name	Span	Value	Min	Max	Average	σ
A	Maximum	Whole trace	503.9 mV	503.9 mV	503.9 mV	503.9 mV	0 V

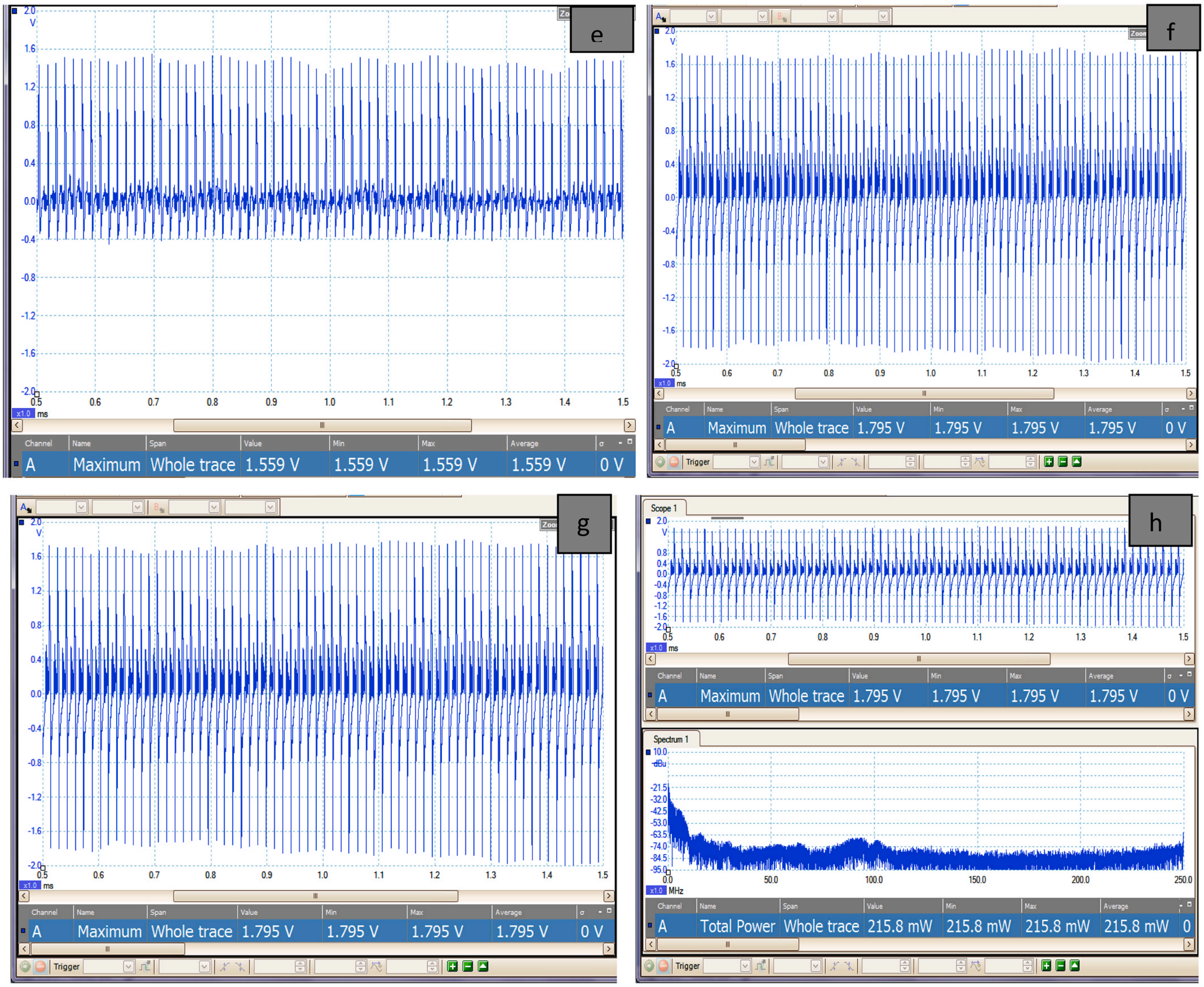
Channel	Name	Span	Value	Min	Max	Average	σ
A	Maximum	Whole trace	1.15 V	1.15 V	1.15 V	1.15 V	0 V

Fig. 5. (a) Output voltage using Au sputtered electrode, (b) Histogram showing occurrence of peak voltage values (c) output voltage generated by ZnO nanowires loaded with CO_2 molecules after 30 min of CO_2 exposure, (d) output voltage after 60 min of CO_2 exposure, (e) output voltage after 90 min of CO2 exposure, (f) output voltage after 2 h of CO_2 exposure, (g) output voltage after 3 h of CO_2 exposure, (h) Maximum output power density of 215 mW/cm^2, after 3 h of CO_2 exposure.

nanogenerator which over all reduced the concentration of free electrons within nanowires and resulted as enhanced piezoelectric potential at output stages.. The effect is well explained by Yuanjie Su [26,39].

Chemisorptions process has occurred, which is being evidenced in output voltage graphs. As the exposure time is increased gradually simultaneously the output voltage is increased because the internal resistance of the ZnO nanowires is increased. Free moving electrons within nanowires are being consumed and reverse leakage current through the nanowires is reduced. Chemiresistive property of ZnO nanowires is related to width of space charge region produced by the adsorption of the CO_2 molecules. Due to adsorption phenomenon depletion layer is created which acted as potential barrier for the conduction band electrons. The process also modulates band bending, as in the oxidizing gases case the band bending is upward as due to depletion of electrons and in *p*-type materials band bending is downward due to accumulation of the electrons. Some results are reported in which various concentrations of CO_2 and additives are used to increase the chemisorptions process [40–42] but the kinetics of the reaction was found quite uncontrollable. In this study, fixed CO_2 molecule concentration 20 ppm has been used but exposure time is gradually increased without using any catalyst to speed up the reaction. For stable and sustained power supply for nanoscale system, the device should be free from such abrupt malfunctions.

Sensitivity phenomenon of various gases on ZnO surface has been reported by many researches around the globe. Some have used additives/dopants [43–45], some have reduced the grain size [46] and some have used variation in temperatures [47] but our finding is unique in this way that no impurity has been used to pure hexagonal wurtzite structures of ZnO nanowires, as by doing so internal dipole creation during the application of external force would be affected and piezoelectric potential may have not produced. Working principle of ZnO based energy harvester is dependent on the generation of piezoelectric potential. In ZnO wurtzite structure O^{2-} and Zn^{2+} are arranged in layer by layer fashion one over the other in balanced condition but when balance is disturbed by an external agent (force) then cations and anions separate with each other hence the creation of dipole in the structure and potential due to this dipole moment is called as piezopotential [48]. Fig. 5(c) indicated 503 mV rise in output voltage after 60min. of CO_2 exposure. Periodic voltage peaks verified that adsorbed CO_2 molecules have enhanced the Edge Effects phenomenon in ZnO nanowires. Edge effects reduced the reverse leakage current and increased the internal resistance of the nanowires. We have reported [7] reduction of reverse leakage current via diameter thinning and its effect on enhanced piezoelectric voltage generation using ZnO nanowires. Afore, mentioned study has enabled us to use pronounced edge effect phenomenon via chemisorptions of CO_2 molecules on ZnO nanowire surface.



Channel	Name	Span	Value	Min	Max	Average	σ
A	Maximum	Whole trace	1.559 V	1.559 V	1.559 V	1.559 V	0 V

Channel	Name	Span	Value	Min	Max	Average	σ
A	Maximum	Whole trace	1.795 V	1.795 V	1.795 V	1.795 V	0 V

Channel	Name	Span	Value	Min	Max	Average	σ
A	Maximum	Whole trace	1.795 V	1.795 V	1.795 V	1.795 V	0 V

Channel	Name	Span	Value	Min	Max	Average	σ
A	Maximum	Whole trace	1.795 V	1.795 V	1.795 V	1.795 V	0 V

Channel	Name	Span	Value	Min	Max	Average	
A	Total Power	Whole trace	215.8 mW	215.8 mW	215.8 mW	215.8 mW	0

Fig. 5. (*continued*).

Chemisorbed CO_2 molecules accumulating on outer surface of nanowires were found time dependant as well. As the exposure time is increased to 90min. output voltage of 1.559V is achieved. Gradual rise in the output voltage has indicated ZnO surface is being modified by CO_2 molecules. Maximum 1.795V with maximum power density of 215.8 mW/cm^2 is observed after 2 h CO_2 exposure. Chemisorbed ZnO nanowires have been exposed for 3 h as well but there was no significant voltage rise was observed. It confirmed that maximum voltage has been saturated after 2 h of CO_2 exposure. ZnO nanowires modified with CO_2 molecules at room temperature for very first time. There could be a tendency of high adsorption of CO_2 molecules at elevated temperatures but at the same time stability of the device could be compromised. At elevated temperatures there are abrupt fluctuations in output voltage, this might be due to desorption process taking place on ZnO nanowires surface. There are various results reported to enhance the sensitivity of ZnO nanowires for gas sensing purposes like Chen et al. [49] used Au functionalized ZnO nanowires but our study is quite unique that no additives are used to enhance the sensitivity of ZnO nanowires but high piezoelectric potential has been achieved by surface modification.

Sheng et al. [50] reported high output voltage of 1.26V by using lateral integration of ZnO nanowires by applying strain of 0.19%. In lateral integrated nanowire structure, there is always a big chance of the nanowires to get plucked out from substrate and lost the connection with electrodes during the external strain. Structure is not very suitable for a long time sustained power supply. VING structure is more stable and robust as compare to LING. Suo et al. [44] designed a two dimensional woven nanogenerator to harvest energy from the environment like wind and etc. the fabrication was carried out by using two fibers woven on small wood and slider which worked as substrates and four pieces of electrodes are used to connect woven nanogenerator to external circuit. It produces an output voltage of 1.2 mV by small airflow. Woven structure is found quite complex to fabricate and it produces a tiny voltage. We reported here quite competitive results as compared to previously reported results. Facile aqueous route for synthesis of ZnO nanowires without using any catalyst and dopant has been used. The output voltage (1.795V) is quite high as compared to above mentioned results. ZnO nanowires based nanogenerators loaded with CO_2 molecules are ideal for future self powered nanoscale systems.

4. Conclusions

High out piezoelectric potential has been generated by ZnO nanowires modified with CO_2 molecules. ZnO based VING structure has been thoroughly examined in CO_2 atmosphere at 20 ppm. Gradual rise in output voltage has been observed during a series of time exposures ranging from 15min. to 3 h. Picoscope graphs verified that ZnO nanowires were fully loaded with CO_2 molecules in 2 h of time. Maximum output voltage of 1.795V with maximum power density of 215.8 mW/cm^2 is being achieved. No dopants are used to enhance the sensitivity of ZnO nanowires. Vertical orientation of ZnO nanowires is being evidenced in XRD pattern. Strong Sharp peak along (002) plane was observed. ZnO nanogenerators modified with CO_2 molecules are viable and sustainable power source to all micro/nanoscale systems where continuous energy is required for longer time.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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