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Adsorption effect of NO_2 on ZnO (100 nm) nanowires, leading towards reduced reverse leakage current and voltage enhancement

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Abstract. Here, we report the adsorption effect of NO_2 on ZnO (100 nm) nanowires. We have studied the effect of adsorbed NO_2 molecules on ZnO nanowire-based energy harvester for an exposure time of 1, 2, 3, 4, 5 and 6 h in a sealed chamber at 50 ppm which yielded piezoelectric voltage of 543.6 mV, 834.6 mV, 1.071 V, 1.78 V, 1.969 V and 2.835 V, respectively. We have thoroughly investigated the behaviour of ZnO nanowires in the presence of NO_2 and observed a maximum output piezoelectric voltage of 2.835 V with a power density 158.2 mW cm^{-2}. This is the first time that ZnO-based piezoelectric energy harvester is being used for the voltage enhancement in the presence of NO_2. We have used vertically integrated nanowire generator (VING) structure. X-ray diffraction pattern revealed the growth orientation of ZnO nanowires were along the *c*-axis from the substrate. ZnO nanowires were grown on indium tin oxide-coated polyethylene terephthalate substrates *via* a hydrothermal route. Surface morphology has been examined by scanning electron microscopy images and diameter of ZnO nanowires was found to be around 100 nm. Piezoelectric voltage has been generated by the VING by applying minute external force of $\sim$50 nN. Periodic output voltage peaks were being measured by picoscope 5204.

Keywords. Piezoelectric potential; VING; nanogenerator; adsorption of NO_2.

1. Introduction

In the era of nanotechnology, there are three most promising nanostructures, silicon nanowires [1–3], carbon nanotubes [4–10] and ZnO nanowires [11–13]. ZnO nanowires exhibiting both piezoelectric and semiconducting properties have been extensively used for nanogenerators [14,15], self-powered nanodevices, biomedical applications [16] and in gas sensors [17,18].

ZnO in its wurtzite structure shows a lack of charge symmetry which generates a piezoelectric potential within the crystal. Piezoelectric phenomenon is vital to use ZnO wurtzite crystal as mechanical transducers, nanogenerators and gas sensors [19–21]. Zn^{2+} and O^{2-} are arranged in a layer-by-layer fashion along the *c*-axis in a hexagonal unit cell. In the absence of an external force, charges are aligned in a balanced position but as the balance is disturbed by an external agent, charge symmetry is disturbed and cations and anions moves apart to create an electric dipole. Electric dipole created piezoelectric potential that lasted only as long as an external force was present. We have used a vertically integrated nanowire generator (VING) structure in our earlier studies to exploit the piezoelectric potential for ZnO-based nanogenerators or energy harvesters [13,15,21]. Schottky contact is an essential part of the VING structure. VING couples both piezoelectric and semiconducting properties. As the piezoelectric potential exists vertically along the nanowires, with an external force there is transient flow of electrons through an external circuit. Schottky contact at one end of VING creates a barrier that ensures the flow of current through an external circuit and stops the current flow back through the nanowires. While an external stress negative potential is developed at the top end of the structure and a positive potential at the bottom end results in current flow through an external circuit from top end to the bottom end. The flow of electrons will not stop, unless all the piezoelectric potential screens out and it will appear as a positive peak in the output stages and as the external force is

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removed, the piezoelectric potential within nanowire diminishes and all the accumulated electrons at the bottom end flow back through the external load, appearing as a negative peak at output stages [22–26].

The study has been carried out by using ZnO nanowires modified with NO_2 molecules for enhanced piezoelectric voltage generation. So far ZnO nanowires have been studied for NO_2 sensing purposes but it is the first time that surface-modified ZnO nanowires are used to generate high output piezoelectric voltage. The study shows a unique perspective to generate high voltage rather than the well-known gas sensing purposes. ZnO nanowires (modified with NO_2 molecules) based nanogenerator could be used as a power source in various nano/microscale systems. High output voltage (2.835 V) generated by a VING which is almost double the AA battery voltage (1.5 V) is quite promising. Several ZnO-based nanogenerators could be integrated (in series) on a small scale chip to produce considerably high voltages which could not only trigger micro/nanoscale system but also sufficient for macrolevel electronic appliances.

In this work, we have studied the effect of adsorbed NO_2 on ZnO-based VING structure. ZnO nanowire structure intrinsically exhibits surface defect (oxygen vacancy) and is oxidized at room temperature $O_2 + e \rightarrow O_2^-$ but NO_2 shows electron affinity (2.273 eV), more than oxygen (1.46 eV) [27–29]. As NO_2 adsorbs on ZnO nanowires O is removed from ZnO surface and NO_2 due to larger electron affinity traps more free electrons in the nanowires, which reduce reverse leakage current through the nanowires and enhance piezoelectric voltage. NO_2 molecules trapping free electrons within nanowires form ions which create an enhanced depletion layer causing high resistance for electron mobility within nanowires reducing reverse leakage current [30]. We have exposed VING structure for a time range starting from 1 h up to 6 h at room temperature and recorded a maximum voltage of 2.835 V with an output power density of 158.2 mW cm^{-2}. A gradual rise in piezoelectric potential for a longer time exposure is due to a large surface to volume ratio of ZnO nanowires. NO_2 molecules adsorbed on long nanowires took time to cover the whole surface defects present on them, and further exposure did not produce any change in the voltage indicating that all the sites have been captured by NO_2 molecules.

2. Materials and methods

We have used all the reagents of analytical grade without further purification. We have grown ZnO nanowires by our earlier reported facile aqueous growth method [15]. In the first step, indium tin oxide (ITO) coated polyethylene terephthalate (PET) substrates are cleaned ultrasonically by deionized water and acetone for 15 and 15 min respectively, and dried in air for 30 min. A volume of 0.1 M molar solution of zinc acetate dihydrate [$Zn(CH_3COO)_2{\cdot}2H_2O$] is used to grow a seed layer on ITO-coated PET substrates. Afterwards, substrates were annealed at 50°C for 1 h.

In the second step, equimolar solution (nutrient solution) of hexamethylenetetramine [$C_6H_{12}N_4$] and zinc nitrate hexahydrate [$Zn(NO_3)_2{\cdot}6H_2O$] was prepared in deionized water. Substrates were immersed in the nutrient solution upside down for 2 h 15 min at 90°C. Nanowire growth is found closely related to growth time in the nutrient solution and the density of the ZnO nanowires was found dependent on the concentration of the reagents.

In our VING structure, the PET substrate acted as a bottom electrode, ZnO nanowires as intermediate part and gold-sputtered electrode as an upper electrode. Emscope SC500 was used to sputter Au electrode on top of ZnO nanowires. Ar pressure inside the chamber was 0.1 Torr; 2 kV is operating voltage of the unit to start charge irradiation process. Au thickness is being controlled by the following relation keeping all values fixed except time which is being controlled by the timer installed on the unit:

$$d = \text{mA} \times \text{kV} \times T \times K, \tag{1}$$

where d is the thickness of the film in Angstroms, mA is current in milliamperes, kV is high tension voltage, T is the sputtering time and K is constant (2 for air and 5 for Ar). A 2.5 nm thick Au electrode was sputtered to produce a Schottky contact with ZnO nanowires. Afterwards, it was heated at 50°C for 10 min to achieve improved adhesion.

Minute external force of $\sim$50 nN is applied on the structure externally by a light plastic roller to produce piezoelectric potential within nanowires. ZnO-based VING structure is exposed to NO_2 gas in a sealed chamber at 50 ppm for various time intervals from 1 to 6 h. Piezoelectric potential generated by VING is measured by picoscope 5204.

3. Results and discussion

Philips XL 30 has been used to obtain scanning electron microscopy (SEM) images of ZnO nanowires grown on ITO-coated PET substrates. Figure 1a–d shows dense growth of ZnO nanowires. It is clear from SEM images that it is a quasi-array of ZnO nanowires of 100 nm. However, the individual diameter of nanowire has also been calculated by using image analysis software (ImageJ) [31]. The SEM image is selected to calculate the top surface area of nanowires. Calibration of scale is required, as software does not have input about the scale of SEM image after the calibration of the scale. The next step is obtaining the binarization grey image. Using image contrasting feature of the software only top surface of nanowires is captured and rest of the portion is eliminated. Then the radius of selected nanowires is calculated by using the following relation:

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

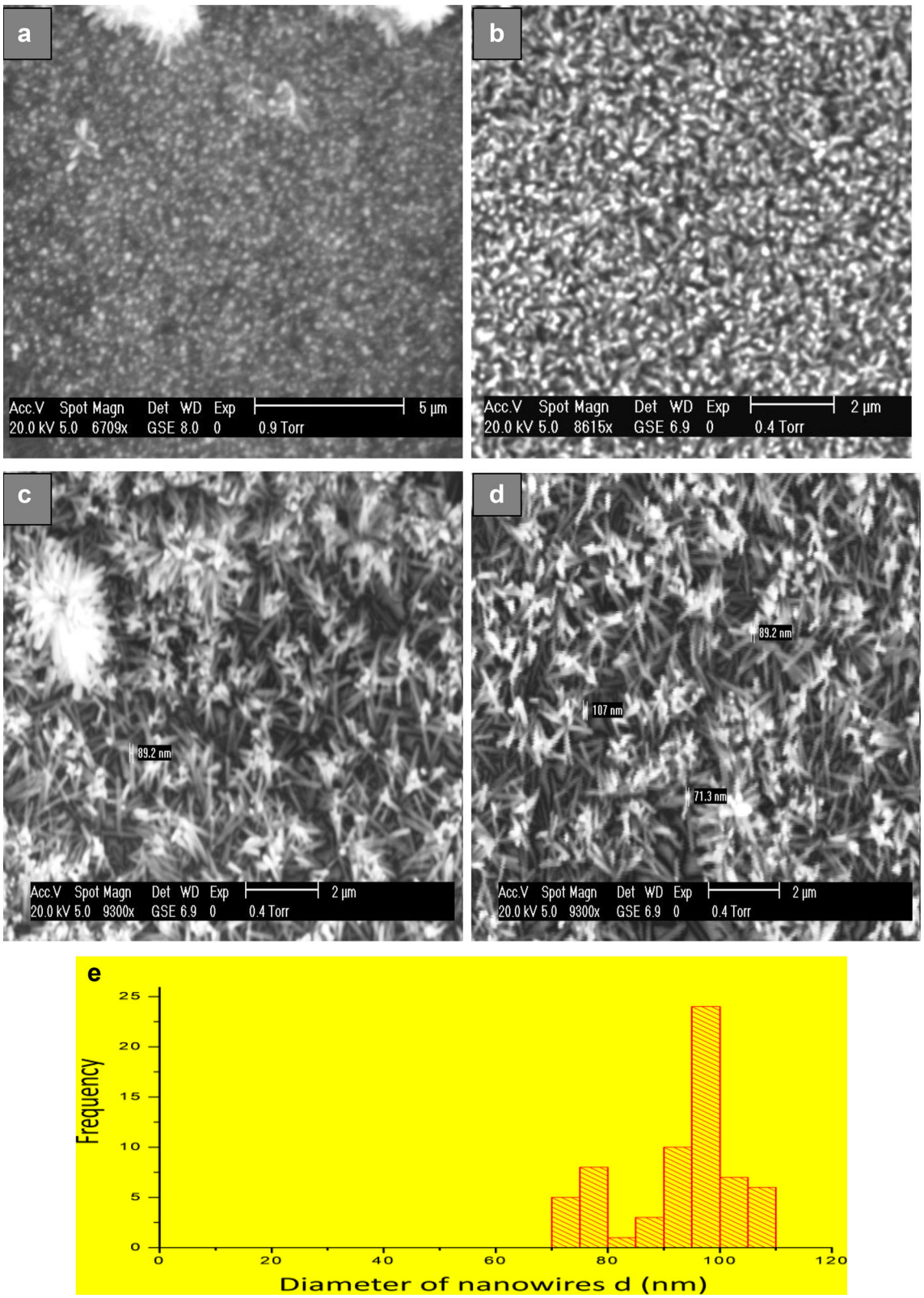

Figure 1. (**a, b, c, d**) SEM images of 100 nm ZnO nanowires grown on PET substrate, at low and high magnification respectively, and (**e**) histogram showing diameter range of nanowires.

Diameter is obtained by multiplying r by 2. The histogram clearly indicated most of the nanowires diameter range is around 100 nm which is exactly in agreement with SEM images.

Density of the ZnO nanowires on PET substrates was controlled by concentration of nutrient solution and diameter of the nanowires was found to be dependent on growth time in the nutrient solution. A narrow opening closer to the top end of the nanowires is necessary for bending during an external force. Our earlier reported values are closely in agreement with our present results [13,15]. Figure 2 presents a schematic view of VING structure and adsorbed NO_2 molecules on the surface of ZnO nanowire.

Figure 3 shows the X-ray diffraction (XRD) pattern of ZnO nanowires. Diffracted peaks from ZnO nanowires at 2θ angle 31.634, 34.4213, 47.4923 and 56.6535 corresponding to crystal planes (100), (002), (102) and (110) are observed. All observed values are compared with standard file (JCPDS 36-1451). It is clear from diffracted pattern that preferred growth orientation of ZnO nanowires is along the c-axis. The prominent peak intensity along plane orientation (002) has verified the argument. Preferred growth orientation of ZnO nanowires along any particular plane could also be determined by texture coefficient (T_c) given by the following relation:

$$T_c(hkl) = \frac{I(hkl)/I_o(hkl)}{1/n\sum n(I(hkl)/I_o(hkl))}, \quad (3)$$

where $T_c(hkl)$ is the texture coefficient, $I_o(hkl)$ is observed intensity peak, $I(hkl)$ standard peak value from JCPDS file and n is the number of diffraction peaks counted. If $T_c(hkl) > 1$ then it shows that preferred growth orientation is along a particular (hkl) plane and if $0 < T_c(hkl) < 1$ then growth orientation is random and it is not along a particular (hkl) plane [32–34]. It is quite clear from the data tabulated in table 1 that majority of ZnO nanowires have a preferred growth orientation along the (002) plane, which is perpendicular to the substrate. T_c values for the prominent peaks along (100), (002), (102) and (110) have been plotted in figure 3b. It is observed that maximum T_c value 1.527 is for the peak along (002) plane and low T_c values are along (100), (102) and (110) planes. Hence, most of the grown ZnO nanowires have preferred growth orientation along (002) plane and very few of them have random orientation which is well supported by SEM images and XRD pattern.

Periodic voltage peaks shown in figure 4a have confirmed the generation of piezoelectric potential within VING that is being recorded by Picoscope. Au/ZnO contact is a Schottky contact. Schottky contact at one end of nanogenerator is essential. In unstrained (no external force) condition, there is no piezopotential within nanowires but as external force is applied the charge centre equilibrium is disturbed and electric dipoles are created resulting in piezoelectric field within nanowires. Negative piezoelectric potential tends to build up at the top electrode and consequently, there is a rise in conduction band and Fermi level of upper electrode relative to the bottom electrode and there is a transient current flow through the external circuit from top to bottom electrode. Schottky contact works as a barrier for the electrons to move through the interface which means that it stops the back flow of electrons through the nanowires and ensures unidirectional flow of electrons from top to bottom. So, all electrons tend to accumulate at the bottom electrode rising up the Fermi level until equilibrium is reached and there is no current through the external circuit. As the external force is removed the piezoelectric field inside nanowire collapses and all accumulated electrons at the bottom electrode move back towards the upper electrode *via* external circuit to regain the charge equilibrium but this time the output signal wave form is opposite in direction so there is an alternating voltage peak in the output voltage spectra [22,35]. For an ideal Schottky contact electron affinity of semiconductor must be less than the work function of metal so that it acts as a barrier for the electrons to move through the interface and it ensures that the flow of the electrons is only from the external circuit and not through the nanowires [13,21]. Gold has a work function (5.1–5.4 eV) which is higher than ZnO electron affinity (E_a = 4.5 eV). It has been tested quite carefully that periodic voltage peaks were generated by the VING or not. Furthermore, the output voltage values have been statistically analysed. It has been depicted in figure 4b that 10 cycles

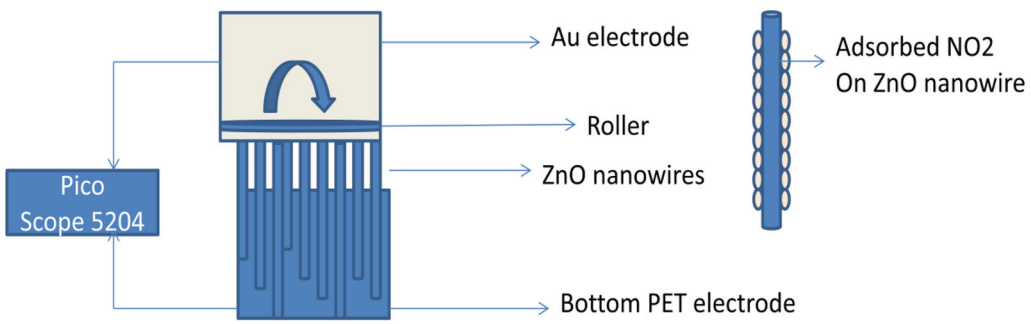

Figure 2. Schematic diagram of ZnO-based energy harvester and NO_2-adsorbed ZnO nanowire.

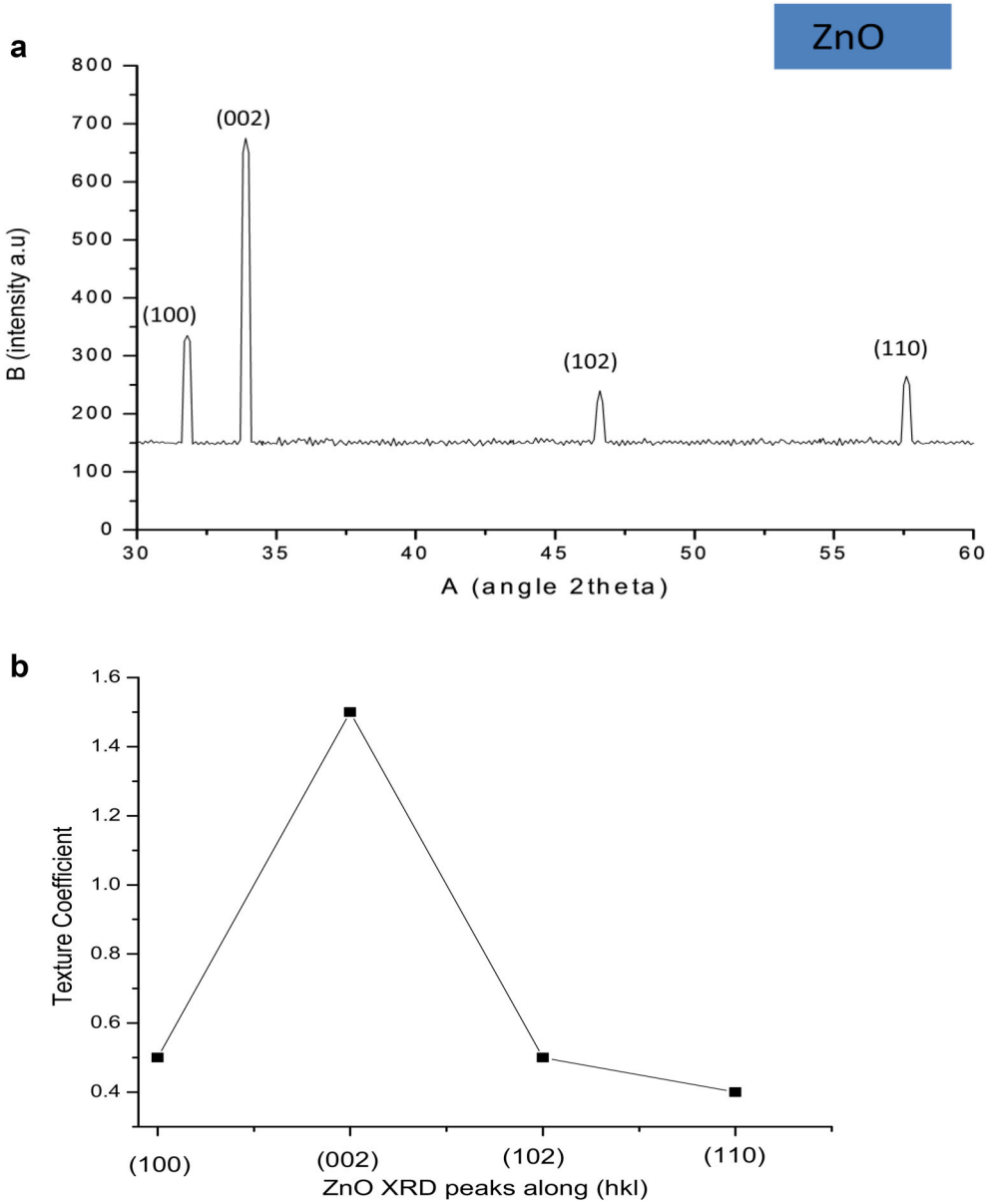

Figure 3. (**a**) XRD pattern of vertically grown ZnO nanowires and (**b**) texture coefficient of grown ZnO nanowires.

Table 1. Comparison of observed XRD pattern with standard JCPDS data.

XRD peaks of ZnO nanowires along (*hkl*) planes	Observed angle 2θ (degree)	Angle 2θ (degree) from JCPDS	Intensity observed	Intensity from JCPDS	Texture coefficient $T_c(hkl)$
(100)	31.6346	31.728	325.112	578	0.562
(002)	34.4213	34.422	675.231	442	1.527
(102)	47.4923	47.494	225.467	229	0.984
(110)	56.6535	56.159	275.392	324	0.849

(forward/backward) were tested to ensure the accuracy of the voltage occurrence peaks. It is clear from histogram that maximum positive voltage peaks were in the range of 0.2–0.4 V and similarly negative voltage peaks were in the range of −0.2 to −0.4 V which is exactly in agreement with periodic voltage peaks shown in figure 4a. To obtain

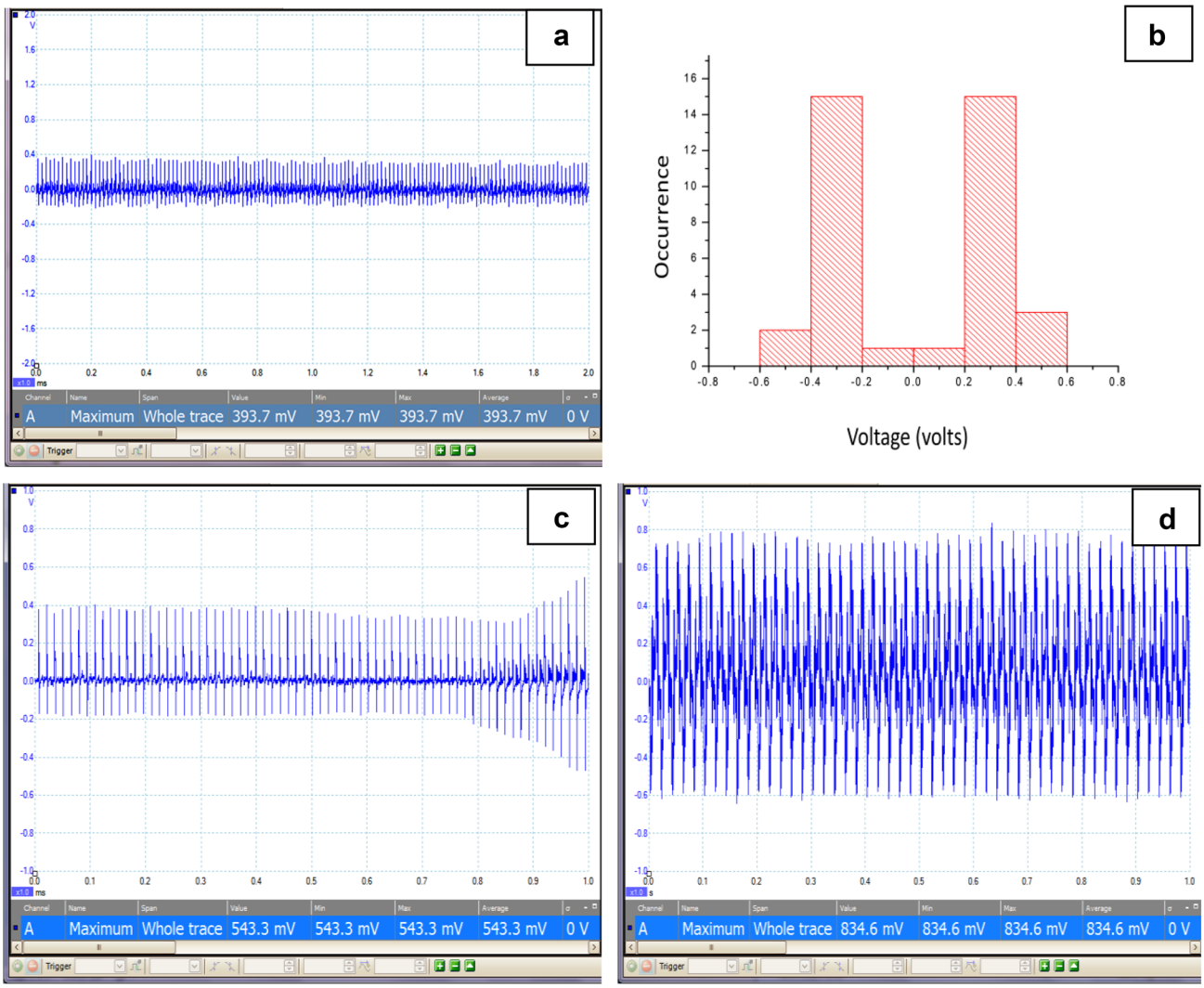

Figure 4. (**a**) Output voltage of 393.7 mV obtained from ZnO nanowires with Au-sputtered electrode, (**b**) histogram showing voltage occurrence for positive and negative cycles, (**c, d, e, f, g, h**) output voltage obtained from ZnO nanowires, after 1, 2, 3, 4, 5, 6 h of NO_2 exposure respectively and (**i**) maximum output power density obtained 158.2 mW cm^{-2}.

voltage occurrence accuracy of VING, the same procedure was repeated after each output voltage measurement. It also rules the displacement hysteresis phenomenon which is generally associated with piezoelectric ceramic actuators in which the external electric field is applied to control the movements and obtain the positioning accuracies. Displacement hysteresis mainly occurs due to ferroelectric effect. Theoretical studies have shown that in piezoelectric ceramics there are electroresistive effects (quite weak) and inverse piezoelectric effect (linear) and energy losses during the domain transition but voltage hysteresis is mainly due to the ferroelectric effect [36,37]. So, it has not been a major concern in ZnO-based nanogenerators.

Figure 4b shows the piezoelectric output voltage after VING is exposed to NO_2 for 1 h in a sealed chamber at 50 ppm. A slight rise in the potential appearing at output voltage 546.3 mV is due to the adsorption of NO_2 molecules on some of the vacant sites (surface defects) available on ZnO nanowires. As NO_2 exposure time is increased to 2, 3, 4, 5 and 6 h enhanced output voltage of 834.6 mV, 1.071 V, 1.78 V, 1.969 V and 2.835 V was observed respectively. It has been observed that voltage value saturates after 6 h of exposure and no further voltage enhancement is seen. A gradual rise in piezoelectric potential is due to clustering of NO_2 molecules on the intrinsic surface defects of ZnO nanowires. Accumulation of NO_2 molecules on long ZnO nanowires has caused pronounced edge effect phenomenon, due to which reverse leakage current through the nanowires is reduced. NO_2 has high electron affinity so it absorbs electrons quickly and gets ionized as shown in equation (4). NO_2 quickly adsorbed on ZnO surface even in the presence of oxygen as shown in equation (5) and it is quite clear from both the reactions that

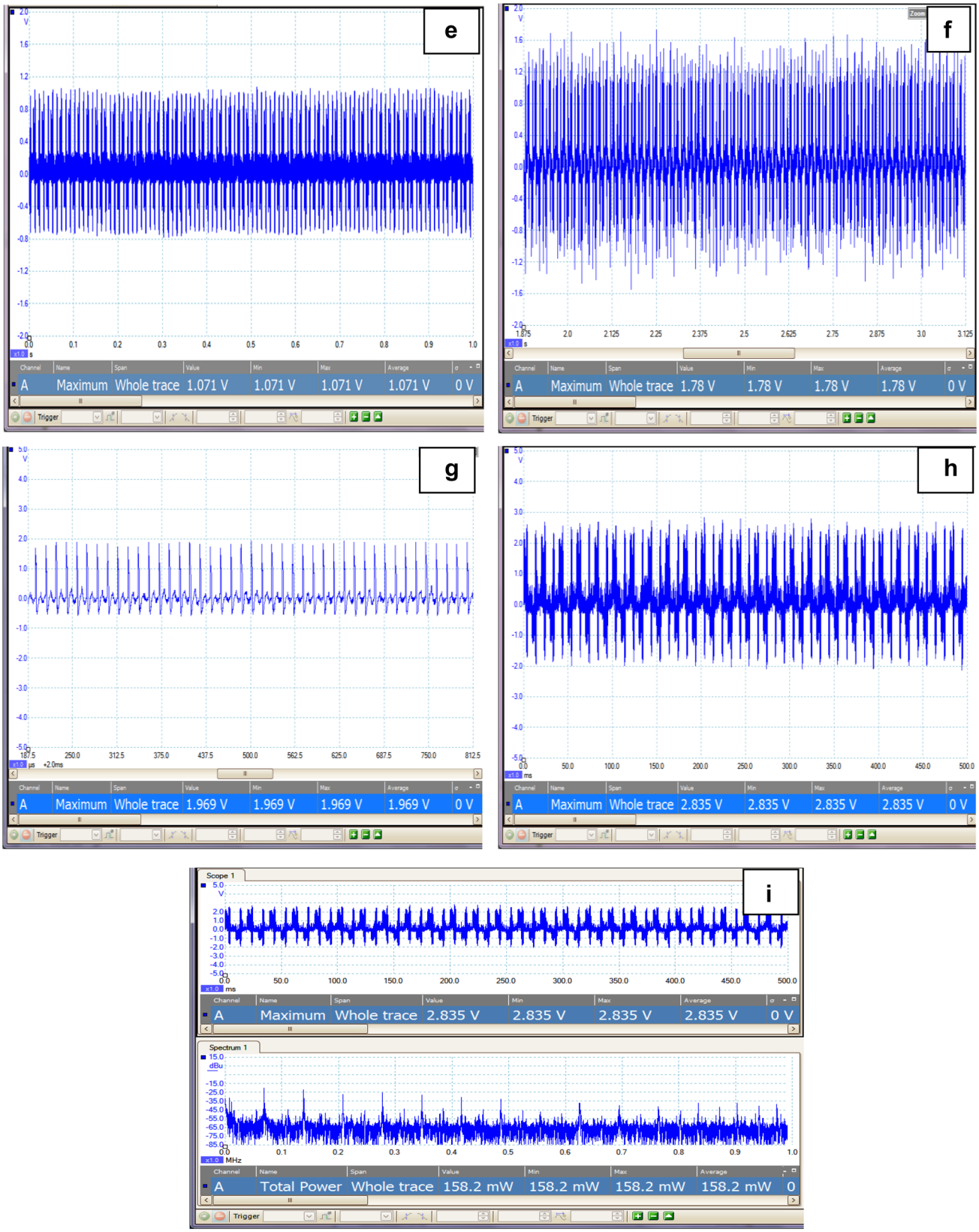

Figure 4. Continued.

NO_2 has reduced conduction band electrons consequently increasing the resistance of nanowires:

$$NO_2 + e^- \rightarrow NO_2^- \quad (4)$$

$$NO_2 + O_2^- + 2e^- \rightarrow NO_2^- + 2O^- \quad (5)$$

As, more and more NO_2 molecules piling up on the surface edges of ZnO nanowires, caused enhanced scattering phenomenon within nanowires. Enhanced scattering phenomenon has increased the internal resistance of the ZnO nanowires and consequently a reduction of leakage current is observed. Reduction in reverse leakage current has been our main objective, and we have achieved it in our earlier study by reducing the diameter range of ZnO nanowires [21]. In this study, we have achieved it by using adsorption effect of NO_2 and as a result of reduction in leakage current, enhanced piezoelectric voltage is being evidenced at output stages. Already very few conducting channels $G = e^2/2h$ [35] are available within nanowires due to smaller diameter which has been further squeezed by the edge effects. NO_2 molecules have higher electron affinity than oxygen molecules [38,39] so it had captured more free electrons moving within ZnO nanowires which further reduced the reverse leakage current and enhanced piezoelectric output potential. We have achieved high output voltage values in the presence of NO_2 at room temperature, as at elevated temperatures of $>150°C$ the desorption process of NO_2 increases which can reduce the output potential. On the other hand at very low temperatures, due to low electron mobility the adsorption phenomenon reduces and consequently piezoelectric potential is reduced [18]. Due to large electron affinity of NO_2 than O, chemisorbed oxygen molecules have been removed from ZnO nanowires by releasing free electrons and NO_2 molecules trapped more electrons transforming to NO_2^- increased the internal resistance of ZnO nanowires which is being evidenced in output piezoelectric potentials.

There are various results reported by using piezoelectric-based nanogenerators, like Yang *et al* [40] reported an energy harvester attached human finger generating output voltage pulses of 25 mV during finger movement. Similarly energy harvester attached to hamster has produced output voltage ranging from 50 to 100 mV. Also by integrating these single nanowire nanogenerators a maximum voltage of 0.5 V was achieved. Yang has used a lateral integrated nanowire nanogenerator structure to grow bio-mechanical energy harvesters which involved a number of complicated and expensive fabrication steps like masking and lithography. Cheng *et al* [41] used VING structure using hazardous material NaOH to grow ZnO nanostructure on conductive polymer and used Au-coated PU film as an upper electrode but the maximum output voltage was 550 mV. Wang and Song [14] reported maximum output voltage of 6 mV during an atomic force microscopy scan on vertically grown ZnO nanowires. Si tip coated with Pt was used in contact mode to produce piezoelectric potential within nanowires, making the technique more expensive. Sheng *et al* [42] reported nanogenerators using VING structure to produce 80, 90 and 96 mV output voltage and also combined linearly to obtain a maximum voltage of 0.243 V. Suo *et al* [43] have proposed two-dimensional woven nanogenerators in fabric which produced output pulses of 3 mV. It is a wearable woven nanogenerator which has used small frictions during fabric movement and converted them to electrical signals. It is quite a complex structure, as the ends of the fibres were attached to strip electrodes and these electrodes are connected to external Cu wires. Pd layer was used to produce a Schottky contact at the intersection of two fibres which itself was quite complex to handle.

In comparison with the abovementioned various results, our results are quite competitive, as facile aqueous growth mechanism does not involve any complex experimental steps, does not require high temperature and vacuum conditions and is cost effective and catalyst free. We have achieved considerably high piezoelectric voltages in the presence of NO_2 under mild conditions. Earlier [13], we have reported output piezoelectric potential of 1.34 V by using 100 nm ZnO nanowires, now we have improved our recent results having a significant rise of 1.43 V.

4. Conclusions

High output piezoelectric potential of 2.835 V with maximum power density of 158.2 mW cm^{-2} is being reported by using 100 nm ZnO nanowires in the presence of NO_2 at 50 ppm. ZnO nanowires have been exposed to NO_2 for various time intervals from 1 to 6 h in a sealed chamber. A gradual rise in output voltage was observed up to 6 h of NO_2 exposure and after that voltage saturation was observed, indicating that all the intrinsic surface defects present on ZnO nanowires were captured by NO_2 molecules. Surface morphology was examined by SEM images and vertical growth is being verified by XRD pattern. We propose high output nanogenerators for self-powered nanoscale systems.

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