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A novel investigation on carbon nanotube/ZnO, Ag/ZnO and Ag/carbon nanotube/ZnO nanowires junctions for harvesting piezoelectric potential on textile

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In the present work, three junctions were fabricated on textile fabric as an alternative substrate for harvesting piezoelectric potential. First junction was formed on ordinary textile as (textile/multi-walled carbon nanotube film/zinc oxide nanowires (S1: T/CNTs/ZnO NWs)) and the other two were formed on conductive textile with the following layer sequence: conductive textile/zinc oxide nanowires (S2: CT/ZnO NWs) and conductive textile/multi-walled carbon nanotubes film/zinc oxide nanowires (S3: CT/CNTs/ZnO NWs). Piezoelectric potential was harvested by using atomic force microscopy in contact mode for the comparative analysis of the generated piezoelectric potential. ZnO NWs were synthesized by using the aqueous chemical growth method. Surface analysis of the grown nanostructures was performed by using scanning electron microscopy and transmission electron microscopy. The growth orientation and crystalline size were studied by using X-ray diffraction technique. This study reveals that textile as an alternative substrate have many features like cost effective, highly flexible, nontoxic, light weight, soft, recyclable, reproducible, portable, wearable, and washable for nanogenerators fabrication with acceptable performance and with a wide choice of modification for obtaining large amount of piezoelectric potential. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4890306>]

I. INTRODUCTION

Nowadays harvesting piezoelectric potential from ambient mechanical energy has widely attracted the research community as an effective energy resource.¹ The idea of energy harvesting from mechanical energy seems more attractive as compared to other resources like thermal energy,² solar energy,³ nuclear energy,⁴ wind energy,⁵ and hydraulic energy.⁶ The idea is to have energy harvested from mechanical resources purely based on the electromechanical coupling of the material that leads to self-power generation.⁷ Therefore, a variety of piezoelectric materials have been tested for harvesting piezoelectric potential by using different techniques. But due to excellent mechanical and piezoelectric properties, ZnO is relatively more attractive as compared to other materials for harvesting piezoelectric potential. Additionally, ZnO is a promising semiconductor and possesses the richest family when considering the variety of its nanostructures. ZnO nanostructures grown vertically have potential for developing self-powered nano-systems. The choice of the substrate is important for the growth of these vertical ZnO nanostructures, and both hard and flexible substrates have been investigated. Textile is the cheapest material which could be used as an alternative flexible substrate with multiple features and applications.^{8,9}

Textiles from natural or synthetic fibers has great potential as a substrate for the fabrication of future energy generation devices due to the various features such as low cost,

abundance, light weight, flexibility, softness, nontoxicity, wearability, and washability. These are some fundamental features of the textile by which we can fabricate flexible, portable, recyclable, and smart nanodevices. In order to use textiles as substrates for such nanodevices they first have to be made conductive in order to collect the generated power. Various conductive polymers such as poly (3, 4-ethylenedioxythiophene) PEDOT and its different derivatives, carbon and its derivatives like carbon nanotubes (CNTs) can be deposited on the textile surface to make it conductive without affecting its fiber architecture. Alternatively, commercially available conductive textile (CT) can be used for such application.^{10–15} CNTs have attracted the attention of the research community owing to their various excellent electrical, mechanical, chemical, and piezoelectric properties. Therefore, CNTs and their derivatives have considerable potential for future generations of devices due to availability in nanoscale with well-defined structures making them suitable for potential application in nanoscale devices. Moreover, CNTs have strong ability of surface modification of different polymers for improvement in their properties.^{16–19} CNTs have also been utilized with different inorganic materials such as GaN-CNT, TiC-CNT, Si-CNT, and ZnO-CNT for enhancing their devices performance.^{20–23} In heterojunction of CNT/ZnO, ZnO itself has various excellent electrical, mechanical, and piezoelectric properties. Moreover, it is also known as a green material due to its nontoxicity. It is a promising semiconductor which can be grown on any surface in a variety of nanostructures like nanorods (NRs), nanowires (NWs), nanoneedles (NNs), nanoflowers (NFs), nanobelts (NBs), nanoflakes (NFKs), nanotubes (NTs), etc.²⁴ All such ZnO

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nanostructures have largely been used for fabrication of various electrical and mechanical devices. Especially on account of the large piezoelectric coefficient of ZnO, it has been employed for harvesting piezoelectric potential by using different techniques. The method of synthesis for obtaining ZnO nanostructures is also very important. Due to the nontoxicity and the low temperature used during growth; the aqueous chemical growth (ACG) has been found to be very suitable and makes it possible to grow different ZnO nanostructures in a few hours.²⁵ To avoid the hazardous chemicals, it would also be beneficial to use aqueous suspensions of CNTs in the fabrication of nanodevices. However, CNTs are not water soluble in their pristine state and form large aggregates in aqueous solutions which are difficult to separate. A commonly used method of dispersing CNTs in water is to add a surfactant which increases the solubility. However, the surfactant is difficult to remove after the removal of the solvent, and the conductivity of the CNTs in the dry state is reduced. Another route to increase the water solubility of CNTs is to attach charged groups to the surface of the tubes. This can be done by oxidizing the CNTs with concentrated acid which results in the formation of carboxyl groups (COOH-).²⁶ Literature reviews disclosed that no comparative investigation about piezoelectric properties of CNT/ZnO heterojunctions by using CT fabric as substrate has been published. Therefore, T/CNT/ZnO NWs, CT/ZnO NWs, and CT/CNT/ZnO NWs junctions were formed for harvesting piezoelectric potential.

In this investigation, ZnO NWs were grown on three samples by using the ACG method. Prior to the growth of ZnO NWs, multi-walled CNT suspension was deposited on samples S1 and S3. After the growth of ZnO NWs, all three samples were used for harvesting piezoelectric potential by using atomic force microscopy (AFM) in contact mode.^{27,28} Surface analysis and the crystalline quality were investigated by using the scanning electron microscopy (SEM), transmission electron microscopy (TEM), and x-ray diffraction (XRD) techniques.

II. EXPERIMENTAL

A. Preparation of CNT suspension

Multi-walled carbon nanotubes (more than 95% carbon, 6–9 nm $\times$ 5 μ m) were purchased from Sigma Aldrich, Sweden.

The nanotubes were added to a mixture of concentrated sulfuric and nitric acid with the volume ratios (3:1) and a total volume of 100 ml. The resulting dispersion was treated in an ultrasonic bath for 1 h to break the nanotube aggregates. After 19 h, 40 ml of concentrated hydrochloric acid was added to the CNT dispersion. Finally, the acid mixture was neutralized with sodium hydroxide solution. The CNT suspension was diluted with deionized water to a volume of 500 ml and was then filtered with a 0.22 μ m filter paper. The filter paper was put in deionized water which was then treated in an ultrasonic bath to re-dissolve the CNTs. The final suspension had a concentration of 1 mg/ml and remained stable for several months. The suspension has no surfactant and therefore has higher conductivity.²⁶

B. Growth of the ZnO NWs

A piece of common textile S1 with a size of 1 cm $\times$ 2 cm (non-conductive) was taken and two other pieces of conductive textile (S2 and S3) coated with silver (ArgenMesh fiber: purchased from Less EMF, Inc., USA) in same size were cleaned by using acetone, isopropanol, and deionized water consequently for several minutes to avoid unwanted contaminations and particles from the surfaces. A layer of suspension containing CNTs was deposited on two samples (1: on non-conductive and 2: on conductive textile) by using spin coater at a speed of 2000 rpm for achieving a homogenous layer of CNTs on the surface of the textile substrates. This process was repeated various times for achieving good conductive and homogeneous layer of CNTs film on surface of the substrates as shown in Figures 1(a) and 1(c). After deposition of the CNTs film, both substrates were heated at a temperature of 70–80 $^{\circ}$ C for 15 min to have a good adhesion of the CNTs film on the sample surfaces. Further, prior to the growth of the ZnO NWs, a homogeneous layer of ZnO nanoparticles was deposited on samples S1, S2, and S3 by using spin coater at the speed of 4000 rpm. This process was repeated three times to achieve a homogeneous and thick layer of ZnO nanoparticles on the surfaces of the substrates. ZnO nanoparticle seed solution was prepared by dissolving zinc acetate and KOH in methanol as given in previously published work.²⁹ Moreover, the seeded substrates were heated again at a temperature of 100 $^{\circ}$ C for several minutes

FIG. 1. Microscopic image of (a) CNTs film coated on a non-conductive textile substrate, (b) after growth of ZnO NWs on CNTs film coated non-conductive textile, (c) conductive textile fabric, (d) textile after growth of ZnO NWs, (e) CNTs film coated conductive textile fabric, and (f) after growth of ZnO NWs on CNTs film conductive textile.

in order to achieve a good seed adhesion on the samples surfaces. After that, all the samples were attached with a Teflon sample holder and dipped into the growth solution, which was prepared by dissolving zinc nitrate and hexamethylenetetramine in 200 ml deionized water in equimolar concentration (0.025M). This container was covered by aluminum foil and placed in an oven at a constant temperature of 90 °C for 6 h. After 6 h, the oven was turned off and the container was left in the oven for several minutes until it cooled down. The samples were then taken out of the solution and cleaned with isopropanol and deionized water, respectively, to remove any unwanted powder or particles from the grown ZnO NWs. Finally, the samples were dried by nitrogen. The samples are shown in Figures 1(b), 1(d), and 1(f).

C. Measurement of the piezoelectric potential

For measurement of the piezoelectric potential AFM instrument manufactured by Netherland (Digital instrument multimode AFM in custom-made trans-impedance amplifier) and a stiff platinum coated probes (NY-MDTNSG 01/Pt) were purchased from Russian Federation were used in contact mode. The instrument was set at fast scanning in the direction of the cantilever to the ground. The cantilever deflection signal was initially set to zero, the set point for the un-deflected cantilever was between 1 V and 5 V, the spring constant was 0.6 N/m, the signal sensitivity was 100 nm/V $\pm$ 20%, and the gain of the trans-impedance amplifier received from the feedback resistor was 1 mV/nA. The input current was less than 25 pA and the product of the gain bandwidth and the AD822 was 1.8 MHz. The resistance of the feedback resistor was 1 M Ω for neglecting the effect of the leakage current with the bias voltage was set at less than 0.5 mV. The samples were

kept at ground potential by a feedback loop of the AD822 operational amplifier for controlling the leakage current. An insulated sample holder was used for the measurement of the piezoelectric current. Positive and negative peaks of the generated output potential were observed from the entire circuit which shows that the current is flowing from the tip into the sample.^{27,28}

III. RESULTS AND DISCUSSION

Since the structure of a single wire of textile has a curved shape, the growth direction of ZnO NWs is changing but it is always along the c-axis direction with respect to the surface of contact. Figure 2 shows the typical scanning electron microscopic images of the surface of the grown ZnO NWs on samples S1–S3. Figures 2(a) and 2(b) show that the grown ZnO NWs on CNTs film coated non-conductive textile are highly dense and vertically well aligned to the surface of conductive textile fiber. Figures 2(c) and 2(d) display the images of the as grown ZnO NWs on conductive textile fabric substrate. Both figures are showing relatively dense and good alignment of the grown ZnO NWs. Figures 2(e) and 2(f) are showing the images of ZnO NWs grown on CNT coated conductive textile fiber with high growth density. On both samples, the grown ZnO NWs are well aligned in the vertical direction and are highly dense. In general, the grown ZnO NWs on all samples exhibited hexagonal wurtzite architecture with homogeneous length and diameters as shown in Figures 2(b), 2(d), and 2(f), respectively.

Figure 3 shows the high resolution TEM images of grown ZnO NWs and CNTs. Figure 3(a) shows the low magnification image of the grown ZnO NWs with CNTs. It can be clearly seen that the diameter of the ZnO NWs is less than

FIG. 2. SEM images of as grown ZnO NWs on (a) CNTs film coated non-conductive textile fabric in low magnification image, (b) in high magnification, (c) low magnification image of as grown ZnO NWs on conductive textile, (d) high magnification image, (e) low magnification image of ZnO NWs grown on CNT film coated conductive textile, and (f) high magnification image of ZnO NWs grown on CNT film.

FIG. 3. TEM images of ZnO NWs with CNTs (a) low magnification images of a single ZnO NW with a few CNTs, (b) diffraction pattern of ZnO NW showing growth orientation in the c-axis direction, (c) high magnification image of ZnO NWs, and (d) high magnification image of a CNT.

200 nm and the diameter of CNTs is around 6 nm, consistent with values reported in previously published article.³⁰ Figure 3(b) shows the diffraction pattern of the grown ZnO NWs,

FIG. 4. Typical XRD pattern of ZnO NWs grown on CNTs film (blue) coated non-conductive textile (UCT) fabric, XRD pattern of as grown ZnO NWs directly on CT (black), and XRD pattern ZnO NWs grown on CNTs film coated CT fabric (red).

associated to the hexagonal wurtzite structures along the c-axis direction on CNT coated fiber.³¹ Figure 3(c) is showing the high magnification image of the ZnO nanowires. The grown ZnO NWs are single crystalline. Figure 3(d) shows the high resolution image of a single CNT having the diameter of

FIG. 5. (a) Schematic diagram of a single ZnO NW, (b) top view of ZnO NW during scanning by AFM tip, (c) side view of ZnO NW during scanning by AFM tip, (d) physical model of ZnO NW in 3D experiences AFM tip, (e) IV characteristics of sample S1, (f) IV characteristics of sample S2, and (g) IV characteristics of sample S3.

around 6 nm. The diameters of ZnO NWs and CNTs in TEM images are consistent with other reported work.¹⁵

Figure 4 demonstrates a typical XRD spectrum (blue) of the as grown ZnO NWs on CNT coated non-conductive textile fabric substrate (S1). All the existing peaks in the XRD spectrum are designated to the specific crystal planes such as (100), (002), (101), (102), (110), (103), (200), (112), and (201) are associated with the hexagonal wurtzite structure of ZnO NWs.^{29,31} One additional peak associated to CNTs is present in the graph. Another XRD spectrum (black) is showing the diffraction peaks associated with the hexagonal wurtzite structure of ZnO along with two additional peaks associated with silver (Ag). The presence of Ag peaks confirm that the conductive textile contain Ag (S2). Another XRD spectrum (red) of ZnO NWs also consists of all similar peaks associated to the hexagonal wurtzite structure of ZnO with two additional peaks of Ag and one additional broad

peak associated with CNTs. It can be seen that a broad peak belonging to CNTs exist between 23° and 25° and is associated with the (002) planes.³² Similarly, in all curves the most intense peak was the (002) indicated that the growth orientation is along the c-axis.^{27–29,31}

A piezoelectric potential is generated when the ZnO NWs are bent by the AFM tip and the mechanical energy from this action induces an electric field along the direction of applied force due to the lattice deformation in the NWs. The mechanical energy is then transformed into electrical energy when the AFM tip and the NWs are in forward bias.⁸ Figure 5(a) shows the image of a single ZnO nanowire and Figures 5(b) and 5(c) display the top and side views of the ZnO NW while experiences a load applied by the AFM tip. A full mechanism of the electromechanical coupling is presented in a physical model shown in Figure 5(d). The AFM tip is attracted to the ZnO NW and the NW bends along the

FIG. 6. Output potential generated from (a) as grown ZnO NW on non-conductive textile (S1) in 3D, (b) piezoelectric potential from small area of S1, (c) current peaks generated from ZnO NW on S1, (d) as grown ZnO NW on conductive textile (S2) in 3D, (e) piezoelectric potential from small area of S2, (f) corresponding amount of current measured from sample S2, (g) S3: CT/CNTs-ZnO NWs in 3D, (h) piezoelectric potential from small area of S3, and (i) amount of current generated from sample S3, when mechanical energy convert into electric potential energy in a ZnO NW during the scanning by an AFM tip.

direction of applied force. Then, due to stretching between two lattice crystals, an electric field is induced in the direction parallel to the applied force and generates positives and negatives charges. Mechanical energy is then converted into electrical energy as shown in Figure 5(d).^{33–35} Figure 5(e) shows the current-voltage (I-V) characteristics of ZnO NWs synthesized on CNT coated non-conductive textile fabric substrate (S1). Figure 5(f) shows the current-voltage (I-V) characteristics of the as grown ZnO NWs on conductive textile fabric substrate (S2) and Figure 5(g) shows the I-V characteristics of ZnO NWs grown on the CNTs film coated conductive textile fabric substrate (S3). Nonlinearity in all three curves indicate that, the junction between AFM (Platinum coated) tip and ZnO NWs is a Schottky junction.³⁶ The difference in the amount of current magnitudes in all I-V characteristics might be due to the surface roughness of the non-conductive and conductive textiles. Other reasons might be the variation in the resistance or it might be due to a difference in the surface bending of the ZnO NWs on conductive textile and on CNTs coated textile. Material properties of the CNTs might also influence the performance, and so giving rise to the difference in the I-V curves with and without CNTs.³⁷

Figure 6 demonstrates a comparison between the amounts of generated output potentials from different nanogenerators fabricated on samples S1, S2, and S3, respectively. Figure 6(a) demonstrates the output potential peaks in 3D. The Pt coated AFM tip was scanned over an area of $10\ \mu\text{m} \times 10\ \mu\text{m}$ of the ZnO NWs and the generated output potential was recorded. Figure 6(b) shows the line profile of the generated output potential from sample S1 in a small selected area.²⁷ Figure 6(c) displays the amount of current generated from the ZnO NWs directly grown on the CNT film coated textile substrate. Figure 6(d) shows many sharp peaks in 3D in order to show the amount of piezoelectric potential generated from the as grown ZnO NWs on conductive textile fabric substrate (S2) in an area of $10\ \mu\text{m} \times 10\ \mu\text{m}$. Figure 6(e) presents the voltage profiles at a selected area of the sample. This figure is showing the piezoelectric potential which was recorded during the scanning over the ZnO NWs by AFM tip in the specific area. Figure 6(f) expresses the profile of the generated current from ZnO NWs grown on conductive textile substrate. Figure 6(g) is showing the 3D images of the generated piezoelectric potential from ZnO NWs grown on sample S3.²⁸ Figure 6(h) shows the line profile of the generated output potential from a selected area of the sample. The graph is showing the piezoelectric potential recorded during the scanning over the ZnO from sample S3. Figure 6(i) depicts the corresponding current peaks captured during the scanning over the ZnO NWs and CNTs junction on conductive textile substrate. The amount of piezoelectric potential from sample S3 is more than the amount of potential generated from samples S1 and S2. The variation in amount of generated output voltages indicates that the CT/CNTs/ZnO NWs junction has good performance as compare to T/CNTs/ZnO and CT/ZnO junctions on textile. It is also confirmed in previously published work that CNT can enhance the device performance.³⁸

IV. CONCLUSION

The aqueous chemical growth method was used to obtain ZnO NWs on plain (S1) and conductive (S2 and S3) textiles fabric to be used as an alternative substrate for piezoelectric nanogenerators fabrication. The surfaces of plain and conductive textiles were modified by CNT film deposition before the growth of the ZnO NWs. Structural characteristics show that the grown ZnO NWs are single crystalline and oriented along the c-axis direction with hexagonal wurtzite structure. The amount of the piezoelectric potential generated from the CT/CNTs/ZnO NWs was relatively larger than the other junctions (CT/ZnO NWs and T/CNTs/ZnO NWs). Also the I-V characteristics of the CT/CNTs/ZnO junction showed better performance as compared to the CT/ZnO NWs and the T/CNTs/ZnO NWs junctions. This investigation confirms that modification with CNTs might enhance the performance of the devices. Also, the use of textile as a low cost, flexible, nontoxic, soft, wearable and washable substrate has shown to be a good alternative to other conventional and expensive substrates.

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