Charge-Generating Mode Control in High-Performance Transparent Flexible Piezoelectric Nanogenerators

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In this work, we demonstrate the mode transition of charge generation between direct-current (DC) and alternating-current (AC) from transparent flexible (TF) piezoelectric nanogenerators (NGs), which is dependent solely on the morphology of zinc oxide (ZnO) nanorods without any use of an AC/DC converter. Tilted ZnO nanorods grown on a relatively low-density seed layer generate DC-type piezoelectric charges under a pushing load, whereas vertically aligned ZnO nanorods on a relatively high-density seed layer create AC-type charge generation. The mechanism for the geometry-induced mode transition is proposed and characterized. We also examine the output performance of TF-NGs which employ an indium zinc tin oxide (IZTO) film as a TF electrode. It is demonstrated that an IZTO film has improved electrical, optical, and mechanical properties, in comparison with an indium tin oxide (ITO) film. Enhanced output charge generation is observed from IZTO-based TF-NGs when TF-NGs composed of only ITO electrodes are compared. This is attributed to the higher Schottky barrier and the lower series resistance of IZTO-based TF-NGs. Thus, by using IZTO, we can expect TF-NGs with superior mechanical durability and power generating performance.

1. Introduction

Energy regeneration technologies may meet critical demand in areas where alternatives to fossil fuels are required for environmental protection, and in applications of portable electronics that require high-density energy capacity or complementary energy sources.[1-4] Renewable energy systems using light, thermal, and mechanical sources have been developed to meet these needs.[5-9] Among these, mechanical energy scavengers utilizing piezoelectricity have attracted great attention because they are suitable for a variety of applications ranging from electronic systems to molecular sensors.[10,11] Intensive studies regarding piezoelectric nanogenerators (NGs) have been carried out, demonstrating new applications such as fiber-type energy scavengers, piezotronic strain sensors, and Schottky contact-based nano/biosensors.[12-14] NGs that are driven by lateral bending of ZnO nanowires using atomic force microscope tip scanning[15] and ultrasonic vibration[16] have shown direct-current (DC) charge generation due to the coupled semiconducting and piezoelectric properties of ZnO.[14,15] The key element in such NGs is the placement of a Schottky barrier between the ZnO nanowire and an electrode, by which carriers are accumulated and released. Alternating-current (AC) power generation from stretching or bending of laterally packaged ZnO fine microscale wires and from direct compression of vertically-aligned ZnO nanowires has also been investigated.[17-20] In these cases, the Schottky barrier formed between the ZnO wires and the electrode acts as a gate that prevents the carriers from being transported through the interface between the wire and the electrode, and also leads to the accumulation of charges, thus providing a higher discharge rate. Thus, from previous charge generation behaviors of AC and DC modes, it can be seen that the charge generation behaviors are mainly dependent on the external operating loads, such as ultrasonic vibration for lateral deformation of ZnO nanowires or compressive pressure for vertical deformation of nanowires. Furthermore, it is clear that the Schottky barrier between ZnO and electrodes is critical to the enhancement of the output performance of charge generation from NGs.

Recently, our group has presented the first demonstration of large-scale transparent flexible (TF) NGs that are operated by flexing the device itself, showing DC-type charge generation.[21-23] Such TF-NGs will lead to new types of embeddable energy harvesting technologies and new applications such as deformable mobile electronics or tactile skin sensors. For TF-NGs, ZnO nanorods are grown on a flexible polymer substrate by an aqueous solution method, where the transparency

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can be controlled by the density of the seed layer provided for ZnO growth. Interestingly, it was found that controlling seed density can lead to different ZnO nanorod morphologies during solution-based growth of ZnO.

Here, we report that the charge-generating mode in TF-NGs of the same device structure can be controlled through variation of the morphology of the ZnO nanorods without any use of an AC/DC converter. It is shown, for the first time, that, when the density of the seed layer for ZnO growth is higher, mainly vertically aligned ZnO nanorods are yielded, on which AC-type charges are generated under a pushing load, while tilted ZnO nanorods grown on seed layers with a low density generate DC-type charges under the same external load. We analyze and discuss the mode transition mechanism for the geometry-induced charge generation from TF-NGs under a pushing load. Furthermore, we show that an indium zinc tin oxide (IZTO) electrode with a higher work function and conductivity than indium tin oxide (ITO) is a promising transparent flexible conductor to enhance the charge scavenging performance of TF-NGs. Based on in situ two-probe resistance experiments, we report the electrical and structural stability of IZTO-based NGs under external mechanical loads such as bending or pushing. The resulting enhanced scavenging performance in both AC and DC type charge generation demonstrates the superiority of IZTO-based TF-NGs.

2. Results and Discussion

2.1. Geometry-Induced Mode Transition of Charge Generation

Figure 1 illustrates the growth of ZnO nanorods on a flexible plastic substrate using the aqueous solution method (see Experimental Section).\(^{21–23}\) ZnO nanorods grew with different morphologies depending on the density of the zinc acetate (Zn(CH\(_3\)COO)\(_2\)) seed layer. Specifically, when a seed solution of 0.01 M concentration was spin-coated on a flexible ITO-coated plastic substrate, a seed layer with a low density was formed (Figure 1a(i)), whereas a high-density seed layer was formed from a seed solution of 0.03 M (Figure 1b(i)). After growth, we observed the differing morphologies of the ZnO nanorods. As shown in Figures 1a(ii) and 1a(iii), primarily tilted ZnO nanorods (T-ZnO) were obtained on the low-density seed layer. However, vertically aligned ZnO nanorods (V-ZnO) were obtained on the high-density seed layer (see Figure 1b(ii) and 1b(iii)). Furthermore, we observed that the dimensions of the ZnO nanorods varied according to the density of the seed layer. T-ZnO nanorods on a low-density seed layer exhibited diameters of 80–90 nm and a density of 45 rods mm\(^{-2}\), as shown in Figure 1a(iii). ZnO nanorods with a larger diameters of 100–110 nm and higher densities of 53 rods mm\(^{-2}\) were observed on high-density seed layers, as shown in Figure 1b(iii). We attribute the morphological change of ZnO nanorods to interfacial tension, which strongly depends on various factors such as the crystal orientation of the seed surface and seed density. The high-density seed layer (Figure 1b(i)) is of the more preferred [001] orientation and has more crystallites than the low-density seed layer (Figure 1a(i)), which results in the formation of vertically well-aligned ZnO nanorods with a high density, in accordance with previous work.\(^{24–27}\)

In order to examine the orientation of as-grown ZnO nanorods with different morphologies, we measured X-ray diffraction (XRD) for V-ZnO nanorods and T-ZnO nanorods (see Supporting Information, Figure S1). In general, ZnO shows a preferred orientation in the [001] direction due to the main polarity. Thus, as expected, XRD spectra show that both types of ZnO nanorods are mainly grown in the [001] direction. However, for V-ZnO nanorods, we could confirm that the V-ZnO nanorods produced on high-density seeds are well aligned, whereas the T-ZnO nanorods grown on low-density seeds are tilted.

We also explored the transparency of T-ZnO nanorods and V-ZnO nanorods on ITO/polyethylenesulfone (PES) films. Optical transmittance results of T-ZnO nanorods and V-ZnO nanorods in the visible range are presented (see Supporting Information, Figure S2). We removed the baseline of ITO/PES from the transmission spectra. As a result, T-ZnO nanorods showed higher transmittance than V-ZnO nanorods. Below a wavelength of around 500 nm, the transmittance decreased significantly. In T-ZnO, the decrease may be explained by the scattering of photons by tilted nanorods, while the larger diameter
and the higher density of V-ZnO nanorods might induce lower transparency than that of T-ZnO. It was found that DC-type charge output is generated from ZnO nanorods (i.e., T-ZnO) grown on low seed density layers obtained from the 0.01 M concentration. DC-type output charge generation is based on the coupled effects of semiconducting and piezoelectric properties of ZnO. When ZnO nanorods are subject to an external force, the nanorods are bent and generate piezoelectric potential due to charges induced via the polarization created by ionic charges of lattice ions along the width of the nanorods. A positive potential is produced at the stretched side of the nanorod and a negative potential is induced at the compressed side as shown in Figure 2.

If we were to apply a rigid top electrode on ZnO nanorods, all force directions transferred from the top electrode by a pushing load would be normal to the electrode. Thus, for example, right-tilted nanorods should bend toward the right-hand direction and left-tilted nanorods bend toward the left-hand direction. Based on the previous charge generating mechanism in NGs,[21] DC-type charges would not then be generated from the NGs since compressive sides of bent nanorods would not be in contact with the top electrode. However, in the case of a soft flexible top electrode, the force directions applied by pushing are different. When we push the flexible top electrode, the electrode is also bent, so that nanorods under the top electrode are actually subjected to forces with various directions. Furthermore, some nanorods can undergo buckling. Thus, right-tilted nanorods can bend towards the left-hand direction under left-handed forces and TF-NGs can then generate DC-type charges by pushing (Figure 2b).

When the ZnO nanorods come into contact with the flexible top electrode through the application of an external force, electrons flow from the compressed sides of the ZnO nanorods to the top electrode.[21] During this process, a Schottky barrier between the ZnO and the electrode plays a critical role in enhancing output performance, since the Schottky barrier accumulates free carriers at the interface.[8,21] This indicates that electrode materials with work functions much higher than the electron affinity of ZnO are extremely desirable to fabricate TF-NGs with high output performance.

It is generally expected that Schottky contact formation between ZnO nanorods and ITO is rather weak, considering the work function of ITO and the electron affinity of ZnO.[21] However, in this work, the Schottky barrier can be substantially changed under an external force due to the change of contact geometry. The weak external force (below 0.1 kgf) leading to slight contact formation between the top ITO electrode and the ZnO nanorods resulted in the observation of the typical rectifying behavior in current-voltage (I–V) measurements (not shown). On the other hand, ohmiclike I–V behavior was observed under external forces above 0.1 kgf. Such an energy barrier leads to resistance of electron flow from the top electrode to the nanorods; but electrons are allowed to flow from the nanorods to the top electrode. However, we cannot totally rule out the possibility of the electron flow from the compressed sides of the ZnO nanorods to the bottom ITO electrode, partially by weak Schottky (ohmic-like) contact formation rather than ohmic contact formation between the nanorods and the bottom ITO electrode.

Figure 3a describes DC-type charge generation from a T-ZnO-based TF-NG. Based on the previously proposed mechanism of charge generation from ZnO nanorods, we attribute the DC-type output signal to the presence of mostly tilted nanorods grown on the low-density seed layer. Since the tilted nanorods are easily bent by an external pushing force (under the load of 0.9 kgf), the piezoelectric potential is formed along the width of the ZnO nanorods. Then, piezoelectric induced charges follow DC-type output behavior along the internal and external circuit of the TF-NG. To verify that the measured signal is from the TF-NG rather than the measurement system, we performed switching-polarity tests,[21–23] as shown in Figure 3a. As the current meter was forward connected to the TF-NG, a positive current pulse was recorded by pushing. When the current meter was reversely connected, the current pulses were also reversed, thus demonstrating that the output signal is generated by our device.

We also measured the electrical current from a V-ZnO-based NG. Interestingly, we found a mainly AC-type output current,[28] as shown in Figure 3b. The AC-type current behavior is attributed to the direct compression of ZnO nanorods by an external force (under the load of 0.9 kgf). Considering the geometry of the V-ZnO, vertically well-aligned nanorods are easily compressed by an external pushing force in the direction of the rod length rather than being bent.[19,21] As-grown ZnO nanorods
have a wurtzite structure and a preferred [001] c-axis growth direction. Since the crystallographic alignment of the nanorods indicates that their piezoelectric alignment is a response to external stress, a piezoelectric potential is generated into the ZnO nanorod along the c-axis under uniaxial strain. Therefore, when an external force results in uniaxial strain of V-ZnO nanorods, one side of the nanorods is subjected to a negative piezoelectric potential and the other side to a positive potential. [17, 19, 21]

Depending on the work function of electrodes at the top and the bottom of the TF-NG, the contacts between the electrodes and the nanorods may be of either Schottky or ohmic types. In order to generate a measurable signal above the noise level from TF-NGs, the presence of a Schottky contact at one end of the nanorods is essential because ohmic contacts at both ends lead to no output signal generation. The Schottky contact at the sides of nanorods with negative potential enhances the output signal by preventing electron flow into the ZnO nanorods through the interface (Figure 4b). The piezo-potential induced electrons are then moved via the external circuit and are accumulated at the interface between the electrode and the side of nanorods with positive potential. As the external force is removed (the compressive strain is released), the piezoelectric potential inside the nanorods instantly disappears and the accumulated electrons flow back via the external circuit.

![Figure 3](image_url)

**Figure 3.** Piezoelectric charge-generating behavior depending on ZnO nanorod morphologies. a) T-ZnO-based TF-NG that shows DC-type charge generation. b) V-ZnO-based TF-NG that presents AC-type charge generation. The switching polarity tests (forward and reverse connections) demonstrate that the output signals are from NGs rather than the instruments.

![Figure 4](image_url)

**Figure 4.** Proposed mechanism for AC-type charge generation in V-ZnO-based TF-NGs. a) The as-received V-ZnO-based TF-NG. b) Electrons flow from the electrode contacting at the sides of nanorods with negative potential to the opposite electrode contacting at the sides of nanorods with positive potential through the external circuit under a compressive force. c) The piezo-potential induced electrons are then moved via the external circuit and are accumulated at the interface between the electrode and the side of nanorods with positive potential. d) As the external force is removed (the compressive strain is released), the piezoelectric potential inside the nanorods instantly disappears and the accumulated electrons flow back via the external circuit.

of bending nanorods. Since the output performance of piezoelectric NGs is mainly dependent on the strain of piezoelectric material, the output current density (about 0.8 μA cm^{-2}) of a T-ZnO-based TF-NG is higher than that (about 0.25 μA cm^{-2}) of a V-ZnO-based TF-NG under the pushing force of 0.9 kgf. Therefore, it can be concluded that the output charge generation mode from TF-NGs under a pushing force is changed between AC type and DC type by the morphological geometry of ZnO nanorods grown on a substrate according to the seed density. Our experimental data support the mechanism that the electric charges can be output in two ways: the electron oscillation in the external load without flowing through the nanorod, under the influence of the piezopotential and the presence of a Schottky contact at the top electrode (AC mode) [17, 19] and the electron flow through the nanorod as governed by the Schottky contact (DC mode). [8, 15, 21–23]
2.2. IZTO-Based Superior TF-NGs

ITO is a useful transparent conductor, but its brittleness, low work function (about 4.7 eV), and low carrier density make it unsuitable as an electrode for high-performance flexible electronics. As an alternative to ITO, IZTO is a promising candidate due to its high transparency, high mechanical stability, and high work function of over 5.0 eV. Accordingly, we employed an IZTO film as an electrode in TF-NGs and investigated the resulting optical and mechanical properties. A 200 nm thick amorphous IZTO film was prepared by means of a specially designed linear facing target sputtering (LFTS) system at room temperature. Due to the effective confinement of the high-density plasma between the ITO and IZO targets and the positioning of the substrate position outside of the plasma region, a-IZTO could be deposited on a plastic substrate at a low substrate temperature of less than 50 °C without heating the substrate by the bombardment of energetic ions and neutral particles. The work function of the as-fabricated a-IZTO films was measured as 5.1–5.2 eV through photoelectron spectroscopy analysis, which is higher than the work function (4.7 eV) of ITO.

The optical transparency of our IZTO film is presented (see Supporting Information, Figure S3). Note that the transmittance of the IZTO film in the green region (450–550 nm) is much higher than that of the reference ITO film. Although the transmittance of the IZTO film in the UV region is lower than that of the reference ITO film, the average optical transmittance of the IZTO film in the full visible range is similar to that of a reference ITO sample. The inset of Figure S3 shows that, to the naked eye, IZTO films have a transparency similar to that of reference ITO films.

In order to investigate the mechanical stability of IZTO films in comparison to that of ITO films, we performed in situ mechanical bending tests. As shown in Figure 5a, the sample size for both films was 3 cm in length. To release the stress and strain concentration in the gripping region, we set the initial position at an orientation angle of about 15 degrees. We controlled the bending radius (r) by decreasing the distance between the grippers of the bending machine. The distance was reduced by 1 mm at each step, the electrical two-probe resistance was then measured, and the corresponding r was calculated at each step. At the fifth step (v) with r of 1.25 cm, the ITO film was broken. We were unable to measure any electrical signals from the sample, which means the ITO film was completely cracked (which could be seen by the naked eye). On the other hand, the IZTO film was broken at the eighth step (viii) with r of 0.85 cm. More importantly, it was found that the major damage to the IZTO after the bending radius of around 0.85 cm occurred in the gripping area, not at the center of the film.

In general, crystallization of ITO films occur rapidly at low substrate temperature due to low amorphous/crystalline transition temperature (T/Tm < 0.19, ~150 °C). However, ZnO doped In2O3 could maintain a stable amorphous structure below 500 °C due to a high amorphous/crystalline transition temperature (~500 °C). In case of the IZTO film, the immiscibility of ZnO and SnO2 in In2O3 also leads to a stable amorphous structure. To make crystalline IZTO films, phase separation of ZnO and SnO2 from In2O3 is necessary. However, the kinetics of phase separation of ZnO and SnO2 in the IZTO film are very slow. Thus, the IZTO can maintain a more stable amorphous structure than conventional ITO film, resulting in the better mechanical durability of IZTO. Thus, we could measure an electrical signal for the IZTO film after recovery. This result indicates that the IZTO film provides much better mechanical/electrical stability for TF-NGs in terms of flexibility.

As mentioned earlier, the Schottky barrier in TF-NGs is critical to enhance the output performance. Since IZTO films provide a high work function (about 5.2 eV) and low sheet resistance (33 Ω/square), we could expect improved output performance for IZTO-based TF-NGs relative to ITO-based TF-NGs. Figure 6a shows the J-V characteristics of TF-NGs. The rectification ratio of the IZTO-based TF-NG was more than four times that of the ITO-based TF-NG, demonstrating the more typical Schottky diode characteristics of the IZTO-based TF-NG due to the higher work function of the IZTO film. Figure 6b exhibits the output current density from a T-ZnO-based TF-NG when we applied IZTO as the top electrode instead of ITO. The DC-type current density was improved by about two times by applying an IZTO electrode under the load of 0.9 kgf. V-ZnO-based TF-NGs also showed slightly enhanced output performance with an IZTO electrode (see Figure 6c). We believe that such enhancement is attributed to the higher work function and improved conductivity of the IZTO electrode in TF-NGs. Thus, we confirmed that the IZTO electrode is highly favorable to TF-NGs in terms of mechanical and electrical properties.

3. Conclusions

We demonstrated that mode control of output power generation in TF-NGs between DC-type and AC-type is based on the morphology of ZnO nanorods grown on different seed densities. We found that tilted ZnO nanorods grown on low-density seed layers generated DC-type piezoelectric charges while vertically aligned ZnO nanorods grown on high-density seed layers exhibited AC-type charge generation. By using IZTO with higher work function and improved conductivity, the Schottky
charge generation performance on b) T-ZnO-based NGs and c) V-ZnO-nanorods (see inset) and improved sheet resistance, compared to ITO. The a-IZTO electrode was co-sputtered on a polyethylene terephthalate (PET) substrate at a size of 30 mm × 30 mm. [29–31]

Measurement and Characterization: Field-emission scanning electron microscopy (FE-SEM) measurements were performed using a JEOL JSM 6100 instrument. Transmission spectra were obtained using a Varian Cary 5000 UV-vis spectrometer. The force loading setup is presented in Figure S4. A Keithley 6485 Picomammeter was used for low-noise current measurements in order to detect currents generated by the NG. I–V measurements were carried out using an Agilent 4156A parameter analyzer.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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4. Experimental Section

Fabrication of ZnO Nanorods: ZnO nanorods were synthesized using the aqueous solution method. Zinc acetate dehydrate [Zn(CH₃COO)₂ · 2H₂O, 0.01 M] and 0.03 M dissolved in ethanol (100 mL) was prepared as a seed solution. The seed solution was then spin-coated at 1000 rpm for 60 s. The spin-coated substrate, covered with a ZnO seed layer with low-density (from 0.01 M solution) and high-density (from 0.03 M solution), was dried onto a hot template at 150 °C. ZnO nanorods were formed on the ITO/PES substrate by immersion into an aqueous solution consisting of zinc nitrate hexahydrate [Zn(NO₃)₂ · 6H₂O, 0.025 M], hexamethylenemethaneamine (0.025 M), and de-ionized water (250 mL). Main growth of the ZnO nanorods was undertaken at 95 °C for 3 h. [19–21]

Fabrication of a-IZTO Film: A 200 nm thick a-IZTO electrode was deposited at a low substrate temperature of less than 50 °C without substrate heating. The substrate was located 80 mm away from the common axis of the targets. At a constant DC power of 800 W, which was simultaneously applied to both ITO and IZO targets, an Ar/O₂ flow ratio of 10/0.4 sccm was applied under a working pressure of 1 mTorr. The a-IZTO electrode was co-sputtered on a polyethylene terephthalate (PET) substrate at a size of 30 mm × 30 mm. [29–31]

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Fabrication of a-IZTO Film: A 200 nm thick a-IZTO electrode was prepared by means of a specially designed linear facing target sputtering (LFTS) system at room temperature. For the co-sputtering of the ITO (10 wt% SnO₂ doped In₂O₃ : Samsung corning precision glass) and IZO (10 wt% ZnO doped In₂O₃ : Samsung corning precision glass) targets, both types of targets were placed in parallel facing each other at a distance of 70 mm apart. Due to the effective confinement of the high-density plasma between the ITO and IZO targets and the positioning of the substrate position outside of the plasma region, the a-IZTO electrode was deposited at a low substrate temperature of less than 50 °C without substrate heating. The substrate was located 80 mm away from the common axis of the targets. At a constant DC power of 800 W, which was simultaneously applied to both ITO and IZO targets, an Ar/O₂ flow ratio of 10/0.4 sccm was applied under a working pressure of 1 mTorr. The a-IZTO electrode was co-sputtered on a polyethylene terephthalate (PET) substrate at a size of 30 mm × 30 mm. [29–31]

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[28] Because the ZnO nanorod growth even on the high density seed layer can form tilted nanorods, the pushing pressure can cause bending of the tilted ZnO nanorods. Thus, we could obtain DC-type output signals from V-ZnO NGs, but it was below 10%.