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Signature of Chairman of Department/Institute:

Signature of Dean of College/ Director of Center:
# 2013 NCKU Annual Report

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I. Abstract

In$_2$O$_3$(ZnO)$_n$ superlattice nanowires containing In-O atomic layers inserted between In/ZnO slabs are successfully synthesized by thermal evaporation and condensation method. From transmission electron microscopy analysis, the thickness of the slabs adjacent to each In-O insertion layers is around 7-8 In/Zn-O atomic layers with M-modulated structures in zigzag shape distributed within to relax the lattice mismatch in In/Zn-O slabs. There are two kinds of superlattice nanowires; i.e, the longitudinal or transverse superlattice structure depending on the insertion layers orientated parallel or perpendicular to the axial direction of the nanowires. Thermal conductivity is measured by Joule-Heating on nanodevices containing single nanowires. The In$_2$O$_3$(ZnO)$_n$ superlattice nanowires demonstrate great thermoelectric properties.

In moving forward, we further demonstrate a new structure by coupling homologous In$_2$O$_3$(ZnO)$_n$ with In-doped ZnO into heterojunction belts synthesized by alloy-evaporation deposition, offering multiple functions with strong confined optical emissions and high power factors. Energy-filter secondary electron images reveal a five layer contrast in the width direction of the belts corresponding to In$_2$O$_3$(ZnO)$_n$/In:ZnO/ZnO/In:ZnO/In$_2$O$_3$(ZnO)$_n$, as confirmed by transmission electron microscopy analysis. The indium-doped ZnO channels confined in two sides behave like quantum wells through band alignment to be predominant in the main UV emission at 385 nm, as revealed by cathodoluminescence spectroscopic imaging. More intriguingly, this novel heterostructure provides an synergetic effect in enhancing electron conduction through the indium doped ZnO layer, while impeding phonon transportation through the homologous In$_2$O$_3$(ZnO)$_n$ layer with numerous interfaces, leading to improved power factor.

Piezoelectric nanogenerators (NGs) made of GaN nanowires are systematically investigated as a function of doping concentration, showing the highest output voltage of 80 mV. Results indicate that free carriers would impose a screening effect, which strongly degrades the output performance of NGs. The work provides a crucial guideline in designing a high-output-power AC NG, with the criterion of a nearly depleted semiconductor as the core material. Moreover, we succeed in integrating vertical ZnO nanowire arrays grown on naked $p$-GaN substrates by a low temperature growth method serving as a perfect template for realizing the piezo-tronic and piezo-phototronic effect. The length and diameter of single crystalline ZnO nanowire arrays can be readily controlled. Thereby, the current-voltage characteristics of the $n$-ZnO/$p$-GaN heterostructure upon strain exhibit the expected piezo-tronic effect, which can be projected into piezo-phototronic applications in the near future.

The work progresses demonstrated not only have promised on the exciting further movement in each cell, but shed a light on the successful integration into hybrid cells in this coming year for higher impact.

Key Words: In$_2$O$_3$(ZnO)$_n$ homologous superlattice nanowires, thermoelectric property, heterostructure, nanogenerators, piezo-phototronic
II. Content of Research Proposal

Background and Significance of Research

2-1 Thermoelectric devices on one dimensional ZnO nanostructures

Based on our previous research experiences, we will extend to grow one-dimensional superlattice structures and explore their applications into thermoelectric and piezoelectronic devices. Development of thermoelectric devices has been proposed as one of the most important solutions to solve energy shortage, however, we lack of natural high-efficiency bulk thermoelectric materials due to a conflict demand between high electrical conduction and low thermal conduction. Thereby, for better thermoelectric devices, thermoelectric materials have to be artificially designed. In this regard, materials made into both nanostructures and superlattices have been regarded to be the most efficient methods to increase effective surface or interface area. Thus, we propose to combine superlattice structure into one-dimensional nanowires in this three year proposal to aim for high thermoelectric efficiency with thorough analysis of the dependence of thermoelectric properties on composition and structure of constituent materials. Finally, we should be able to demonstrate the best energy management strategy by integrating thermoelectric devices with other energy-harvest nanodevices, here nanogenerators, as the most high-performance hybrid energy nano-devices to harvest different forms of energies simultaneously. The successful hybrid nano-device will be the high impact one.

Introduction of the thermoelectric properties

To assess the efficiency of a thermoelectric device, a thermoelectric figure of merit is defined as $\text{ZT} = \frac{S^2 T \sigma}{\kappa}$, where $S$ is the Seebeck coefficient; $T$ being the absolute temperature; $\sigma$ being the electrical conductivity and $\kappa$ is the thermal conductivity. To enhance the ZT value of thermoelectric materials, there are generally two approaches:

1. Improve the power factor (power factor = $S^2 \sigma$); which can be affected by four factors: (a) scattering parameter, (b) density of states, (c) carrier mobility, (d) Fermi energy level. The power factor can be optimized by changing doping concentration to adjust Fermi energy level.

2. Reduce thermal conductivity $\kappa = \kappa_e + \kappa_L$; where $\kappa_e$ and $\kappa_L$ are the thermal conductivity of electrons and lattices, respectively. Most thermal conductance of thermoelectric materials is governed by lattice vibrations (phonons), thus reducing $\kappa_L$ becomes important to improve ZT. Since $\kappa_L = \frac{1}{3} C_v V_l$, where $C_v$ is specific heat; $V$ being velocity of sound in the material, and $l$ is the mean free path, which can be altered by impurities and grain boundaries. Therefore, in order to enhance the ZT value by reducing thermal conductivity, we generally reduce the mean free path of phonons by increasing phonon scattering probability, by which only thermal conductivity will be reduced without affecting electrical conductivity. Low dimensional nanomaterials have the characteristic size smaller than phonon mean free path, enforcing phonon mean free path limited by the size. The scattering probability of phonons will be substantially increased, resulting in the reduction of phonon contribution to thermal conductivity.
Thermoelectric properties of one dimensional nanostructure

In addition to reducing the size of the materials, creating more internal interfaces can also be implemented to decrease thermal conductivity. These coherent interfaces will scatter phonons more than electrons. Thus, thermal conductivity can be reduced, while electrical conductivity is kept. Thereby, superlattice structure is one efficient way to enhance the thermoelectric properties. Yang et al have reported ZnO superlattice nanowires [1]. They deposited In and Ga nanoparticles on the sidewalls of the ZnO nanowire arrays by thermal evaporation, followed by annealing under high temperature oxygen atmosphere. In and Ga atoms at high temperatures diffuse into the nanowires through Zn vacancies to form superlattices, and the superlattice structure is composed of InO$_2$-octahedral planes and Wurtzite MZn$_{n}$O$_{(n+1)}$ (M = In, Ga) tablet. Thermal conductivity is significantly decreased, while Seebeck coefficient is increased due to low energy electron being filtered by superlattices. A small amount of In and Ga atoms can also be doped into the ZnO nanowires to enhance electrical conductivity. Therefore, while the conductivity and Seebeck coefficient of the nanowires are increased, thermal conductivity is decreased. Three thermoelectric coefficients are enhanced at the same time, resulting in thermoelectric figure of merit rise by 2.5 orders of magnitude (from 1.7x10^{-4} for ZnO nanowires to 0.055 for the IGZO nanowires at 300 K). It not only shows excellent thermoelectric properties of the 1D superlattice structure but also proves that the structure is one of the important directions in the future thermoelectric field.

Methods for measuring thermoelectric properties

Measuring thermoelectric properties of 1D structures is extremely hard and non-standard. There are two methods developed depending on whether nanowires are in contact with the substrate. In the first approach, the nanowire is placed on a silicon substrate with a SiO$_2$ layer, and the electrodes and the heater are made by electron beam lithography on the substrate. Electrical current into the heater converts to Joule heat, by $P = IV$, to produce a temperature gradient on the substrate. By establishing relationship between resistance and temperature of a material, the temperature gradient, $\Delta T$, can be extracted by measuring resistance at the both ends of the nanowire. At the same time, the voltage difference $\Delta V_P$ between the both ends of the nanowire is also taken, so that Seebeck coefficient can be learned by $S = -\Delta V_P/\Delta T$, and also temperature-dependent thermoelectric power (TEP) at different temperatures. We can apply electric field effect to make carriers injection into nanowires on a highly doped silicon substrate. This method will enhance the conductivity of the nanowire. This device also can measure the variance of resistance at different temperatures.[2] In the second method, the nanowire is suspended from the substrate, and only both ends of the nanowire contact with the electrodes. With this method, thermal conductivity of a substrate will not be counted, and thermal conductivity can be measured by 3ω method.[3] In addition, with regard to the measurement of thermal conductivity, Abstreiter et al. employed micro-Raman to measure thermal conductivity of a single nanowire[4]. The authors focused a micro-Raman laser onto the middle of a suspended GaAs nanowire, which contacts with Au films at both ends. At the laser focal point, there would
be local heating, and heat will be transported from the middle of the nanowire to both ends. Because the thermal conductivity of Au is much higher than GaAs, the heat will be transported rapidly from both ends of the nanowire to Au. Therefore the temperature in the center will be higher than in both ends, resulting in a temperature gradient. Thermal conductivity can be obtained by the relationship between Raman shift of TO peak and temperature.

2-2 Nanogenerator
As the fossil fuel consumes in an incredible rate, the renewable energy becomes an urgent and important issue now in our world. In order to solve the energy crisis problem, utilizing natural energy such as motion, vibration and liquid flow and convert them to electricity become an important breakthrough in achieving self-powered nanosystems. With these self-powered nanosystems, the energy supplying systems can be further miniaturized. Since 2005, the piezoelectric effect and nanogenerator in Wurtzite nanostructures have been investigated. [5] The unique well-aligned property of ZnO nanowires benefits to the energy harvest from mechanical energy to electricity in nanoscale. Simultaneously controlling the applying forces on the ZnO nanowires by atomic force microscopy (AFM) and Schottky barrier height at metal-semiconductor interface, the mechanical energy can be easily transformed to electricity and stored in the nanowires temporarily. Followed by connecting the nanowires to the desired devices, the electricity can be further released. The fundamental of this nanogenerator system is based on the piezoelectric effect, which generates the electricity itself by deforming the crystal structures under external applied forces. There are lots of motion types in our life, for example, the pressure underneath our shoes when we walk, the muscle stretches and the heart beats, and so on. All of these can be harvested and generate electrical energy. Nowadays, the research of nanogenerators is mainly based on semiconductor compounds that are formed in Wurtzite structure and the most common materials are ZnO[6], ZnS[7], CdS[8], GaN[9] and InN[10]. The basic principle of a nanogenerator is the coupling of semiconductor property and piezoelectric effect. Prof. Zhong Lin Wang is the pioneer in this field. They investigated well-aligned ZnO nanowires by AFM equipped with a Pt-coated Si tip under contact mode. The diameter and length of ZnO nanowires are 20-50um and 1-2um, respectively, and the tip force was kept at 5nF. ZnO nanowires bent and recovered during AFM tip scanning and almost half of them induced 3-12mV output voltage. However, this phenomenon was not observed in Si, WO₃ nanowires and carbon nanotubes.

Another key discovery is that the output voltage is induced only when ZnO nanowires are under compressive strain. As a ZnO nanowire is bent by an AFM tip, the ZnO nanowire bends and exhibits tensile strain on one side and compressive strain on the other side. Owing to the ions’ displacement, the ZnO nanowire induces a positive piezoelectric potential under tensile strain, and a negative one under compressive strain. When the Pt-coated Si tip contacts with the tensile side of the ZnO nanowire, the positive piezoelectric potential applies on the Schottky diode formed by Pt and ZnO. Under this situation, the Schottky diode is at a reversed bias state, and no current goes through the diode. On the other hand, when the tip scans over and contacts with the
compressive side, the negative piezoelectric potential acts as a forward bias state to the Schottky diode. Thus, the electrons would flow outside and induce a negative output potential, and this also illustrates that the delay of output voltage and the basic principle of a nanogenerator.

Since the synthesis of ZnO nanowires is well developed, ZnO nanowires become the major material for fabricating nanogenerators. Compared to ZnO nanowires, CdS, ZnS, GaN and InN nanowires are just at its infancy on nanogenerator research and only single nanowire based properties were investigated. Under conductive AFM measurements, single CdS nanowire exhibited 3mV output voltage [8], 2mV for ZnS [7], 20mV for GaN [9] and 110-120mV for InN. Some work manifested that InN exhibited over 1V output voltage. [10]
III. Results and Discussion

3-1 Thermoelectric devices

Highlighted results that are World-leading or Taiwan-leading:

1. **Two types of homologous In$_2$O$_3$(ZnO)$_n$ nanowires with vertical and horizontal superlattices first synthesized**

   Figure 1 shows the FE-SEM image of the as-grown products, consisting of wire- and belt-like structures. Of which, the typical nanowires are characterized by 5-15μm in lengths and 50-120 nm in diameter, which are composed of homologous In$_2$O$_3$(ZnO)$_n$ superlattice structures. Figure 2(a) shows the low magnification TEM image of such a single nanowire. Like the other nanowires, this nanowire has a gold nanoparticle at its tip acting as a catalyst via the vapor–liquid–solid growth mechanism. Figure 2(b) is the HRTEM image, showing modulation contrast along the longitudinal axis, confirming to a In$_2$O$_3$(ZnO)$_n$ superlattice structure. This superlattice structure consists of In-O and In/Zn-O layers stacked alternately along the c axis with the spacing between the In planes and the nearest Zn planes being 0.32nm, which is larger than the Zn (0002) interplanar spacing of 0.27nm. The increase in the d spacing is due to the larger ionic radius of In than that of Zn. In addition, the HRTEM image reveals periodic superlattice structure with identical distance between two In-O layers, being around 7-8 In/Zn-O atomic layers. Therefore, this nanowire can be described by a precisely defined unit cell as expected for the In$_2$O$_3$(ZnO)$_n$ compounds in theory. Moreover, this structure is obviously reflected in Figure 2(c) of the SAED pattern, with a series of satellite spots between two main spots of ZnO.[11]

   By referring to the enlarged HR-TEM, shown in Figure 2(d), M-modulated structures in zigzag shape become evident within each ZnO slabs. Elastic strain can build up in the ZnO layer (with the strain energy increasing as increasing the ZnO layer number $n$) since the ideal in-plane lattice constants of this plane differ significantly from that of ZnO. Hence, the formation of M modulation in the In/Zn-O layer can reduce the total energy of the structure. From the total energy calculation results[12], the total energy of the modulated structure is lower than the structure with the flat-layer In-O boundary. There are reasons to explain why modulated structure is energetically favorable. First, considerable strain will be induced around the In–O for the flat In–O layers inside the ZnO slab resulting from highly mismatched lattices with ZnO. By forming the modulated structure, this strain can be significantly reduced, as In and Zn atoms arrange alternatively. Besides, O atoms are threefold coordinated in the flat In–O layers and becomes energetically unfavorable whereas in the modulated structure, all O atoms in the zigzag boundary are fourfold coordinated, which is energetically more favorable[12]. In addition, the In-O octahedral layer acts as the inversion boundary so that the polarities of ZnO at its two sides are inverted. Consequently, the polarities must be inverted again inside the Zn/In-O slabs. In other words, the zigzag boundaries provide the required second polarity inversion[13].
To confirm the microstructure of the In$_2$O$_3$(ZnO)$_n$ nanowires, Z-contrast STEM imaging is employed as shown in Fig. 2(f), where the intensity is approximately proportional to the square of the atomic number of the constituent atoms. Thus, using Z-contrast imaging, the location of In (Z= 49) can be unambiguously determined, but oxygen cannot be imaged due to its relatively small atomic number[1]. Fig. 2(e) clearly shows the presence of In-enriched layers (bright lines) oriented perpendicular to the [002] direction. As observed in Fig. 2(f), the In atoms segregate on individual planes and are separated by wurtzite In/Zn-O slabs. Only single layers of In atoms are observed, consistent with the layers being composed of octahedrally-coordinated InO$_2^-$, which has been shown to be the most stable configuration for In within the superlattice structure.

Besides the nanowires with a longitudinal superlattice structure, nanowires with transverse superlattice structure have also been observed in the product. As shown in Figure 3(a), layers do not stack along the growth direction [10-10] of the nanowire but along the transverse direction of the ZnO [0001] direction, which is confirmed the corresponding SAED pattern, in Figure 3(b). If we enlarge the Z-contrast image, shown in Figure 3(c), the zig-zag modulation is more apparent than the transverse superlattice structure. However, unlike the longitudinal superlattice structure, the In-O stacking sequence along the c axis is not identical. Therefore, this nanowire cannot be described by a precisely defined unit cell. Lastly, from the Z contrast image shown in Figure 3(d), the spacing between the In planes to the nearest Zn planes is 0.31nm whereas the In/ZnO (0002) interplanar spacing is about 0.27nm.

Figure 1:  FE-SEM image of the as-synthesized In$_2$O$_3$(ZnO)$_n$ nanowires.

Fig.2: (a) Low magnification TEM image of a In$_2$O$_3$(ZnO)$_n$ nanowire. (b) High-Resolution (HR)-TEM image showing different stacking sequences. (c) SAED pattern. (d) Enlarged HRTEM
image from (b). (e) STEM Z-contrast image. (f) Enlarged Z-contrast image.

Fig.3: (a) HR-TEM image of a transverse superlattice structure with different stacking sequences. (b) SAED pattern. (c) Enlarged HRTEM image from (a). (d) Z contrast image.

2. Enhanced thermoelectric properties with the In$_2$O$_3$(ZnO)$_n$ superlattice nanowires

Various nanodevices are first devised to take measurements for thermal conductivity and Seebeck coefficient from individual nanowires. As for nanodevices measuring thermal conductivity, the nanowire is designed to be suspended as shown in Fig. 4 so that heat is ensured to pass only through the nanowire without any excursion through the underlined layer. The heat is self-generated through resistive heating or Joule heating by a constant DC current through the nanowire. Therefore, the heating power of the nanowire can be described as $P = I^2R$. The both gold electrodes are regarded as heat sinks due to inherently excellent thermal conductivity to establish equilibrium temperature with the ambient. Therefore, during Joule heating, heat flows from the highest temperature at the central part of the nanowire toward both ends of the electrodes until a steady state for establishing temperature gradients is reached where the power input through Joule heating is equal to the power output leaving from both electrodes. Meanwhile, to avoid heat dissipation by thermal convection caused by ambient gas molecules in the theoretical derivation, the measurements are carried out under vacuum. Based on this model, thermal conductivity has been derived and given by: [14]

$$\lambda = \frac{mI^2RL}{12A\Delta R}$$

where $m = \Delta R/\Delta T$. $\Delta R$ is determined from the change of the resistance between initial state and steady state during Joule heating. The parameter of $m$ has to be determined independently by reading the resistance value against ambient temperature when heating the nanowire as a whole on a hot plate. The rest of the parameters including length, $L$, and area, $A$, can be measured from the SEM image in Fig. 4. Ultimately, thermal conductivity is calculated by the above formula.
Fig. 5 shows an optical microscopy image of a real device measuring Seeback coefficient by using electron beam lithography process. First, the temperature coefficient of resistance, $\alpha$, of the Ti/Au electrode on the nanowire is measured by four-point probe measurements. Then, a dc current is applied to one of the two micro-heaters to generate heat through Joule heating, establishing a temperature gradient along the nanowire. The temperature of each Ti/Au electrode on both terminal ends can be determined by measuring resistance of each electrode with the pre-determined $\alpha$ value to calculate the temperature difference across the nanowire. The voltage difference across the nanowire is read out directly. Consequently, Seeback coefficient can be determined by sweeping the applied dc current for different heating power. Table 1 compares the measured results between the In$_2$O$_3$(ZnO)$_n$ with pristine ZnO nanowires. Compared to ZnO, both thermal conductivity and Seebeck coefficient of the In$_2$O$_3$(ZnO)$_n$ nanowire are enhanced by introducing superlattice, at the expense of electrical conductivity. Therefore, ZT value of the In$_2$O$_3$(ZnO)$_n$ nanowire is only two times larger than ZnO. We, therefore, come out with another novel microstructure as further improvement in the next section.

Fig. 4: SEM image of a suspended In$_2$O$_3$(ZnO)$_n$ nanowire device allowing to measure the change of resistance after Joule heating by applying a constant current to the nanowire

Fig. 5: OM image of an In$_2$O$_3$(ZnO)$_n$ nanowire device for measuring Seeback coefficient.
Table 1: Thermoelectric properties of ZnO and In$_2$O$_3$(ZnO)$_n$ nanowires

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<th>ZnO nanowire</th>
<th>In$_2$O$_3$(ZnO)$_n$ nanowire</th>
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<tr>
<td>Electrical conductivity ($\sigma$)</td>
<td>19.9 $\Omega^{-1}\text{cm}^{-1}$ (at 296K)</td>
<td>0.33 $\Omega^{-1}\text{cm}^{-1}$ (at 297K)</td>
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<tr>
<td>Seebeck coefficient (S)</td>
<td>-163.72 $\mu$V/K (at 296K)</td>
<td>-361.83 $\mu$V/K (at 297K)</td>
</tr>
<tr>
<td>Power factor ($S^2\sigma$)</td>
<td>5.33x10$^{-5}$ Wm$^{-1}$K$^{-2}$ (at 296K)</td>
<td>4.32x10$^{-6}$ Wm$^{-1}$K$^{-2}$ (at 297K)</td>
</tr>
<tr>
<td>Thermal conductivity ($\kappa$)</td>
<td>14.95 Wm$^{-1}$K$^{-1}$ (at 296K)</td>
<td>0.53 Wm$^{-1}$K$^{-1}$ (at 350K)</td>
</tr>
<tr>
<td>Figure of merit (ZT)</td>
<td>1.05x10$^{-3}$ (at 296K)</td>
<td>2.42x10$^{-3}$ (at 297K)</td>
</tr>
<tr>
<td>$dR/dT$=$m$</td>
<td>-17.15 $\Omega$/K (at 296K)</td>
<td>-2.84x10$^5$ $\Omega$/K (at 297K)</td>
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3. **A novel heterojunction belts by coupling homologous In2O3(ZnO)n with In-doped ZnO for further enhanced thermoelectric properties**

The coupled structure of homologous In2O3(ZnO)n with In-doped ZnO into heterojunction belts is successfully fabricated by alloy-evaporation deposition. This work demonstrates a new epitaxial layered structure by combining doping in one layer and homologous structures in another.

Figure 6(a) is a low-magnification SEM image, showing belts of lower density formed accompanied by higher density nanodisks (NDs) on a silicon substrate, where the red dotted circles mark the nucleation sites of the belts. The belts exhibit a tapered morphology, shrinking from bottom to top, with typical lengths and widths falling in the range of 10-20 $\mu$m and 1-2 $\mu$m, respectively. Figures 6(b) and 6(c) show higher magnification SEM images of the NDs and belts, respectively. The NDs are partially hexagons and partially half-hexagons, with a diagonal size of around 0.8~1 $\mu$m randomly oriented on the substrate. Careful examination of some vertically oriented NDs reveals that their thicknesses are in the range of 60-100 nm, with most having two parallel side edges with a cone on top. Interestingly, the belts with thicknesses in the range of 250~350 nm also show a sword-like shape, with two parallel side edges and a tip of around 60~70° on top. The similar features of these structures, except for the aspect ratio and size, indicate that the belts grow longer, extending from nanodisks as embryos.

The SE images acquired from the lower and upper detectors at an accelerating voltage of 1 kV with a working distance of around 3 mm are shown in Figures 6(d) and 6(e), respectively. The image from the lower detector is obtained by collecting both back-scattered and secondary electrons, and is an unfiltered image for greater topographic contrast. In contrast, the upper detector can only collect SEs up to a certain energy, depending on the bias applied to the side electrodes, forming a low-pass energy filtered image for voltage contrast, which is sensitive to the electronic structure, as dictated by parameters such as doping concentration.[15] Figure 6(d) reveals no clear change in contrast throughout the belt, as also indicated in the inset for the intensity line profile projected from the marked white rectangular area. The near constant
contrast implies that the belt is characterized by a flat surface morphology. In contrast, Figure 6(e) exhibits a distinct sandwich-like contrast for the belt, indicative of being composed of a heterojunction layered structure. As revealed in the projected intensity line profile in the inset, two dark bands are sandwiched by the bright central area and two bright bands at either side, forming a five-layer structure, where the two brightest lines at the edges due to edge effect are excluded. The symmetric five-layer structure can also be seen in a higher magnification SEM image in Figure 6(f), which clearly shows a heterojunction structure rendered by non-uniform In doping. In order to confirm this conjecture, Figures 6(g)-(i) show EDS elemental maps of indium, zinc, and oxygen, respectively, with reference to the SEM image in Figure 6(f). Intriguingly, these three elements demonstrate drastically different spatial distributions. Whereas oxygen is uniformly distributed over the entire structure, In is richer at the bottom and two sides covering from the area corresponding to the side bright bands, and perhaps extending to the two dark bands in Figure 6(f). Meanwhile, zinc seems to decay slightly and symmetrically toward the two sides, and probably also in the two bright bands. These results reveal the complex dependence of indium doping on the five heterojunction layers through the influence of the electronic band structure, responsible for the strong SE contrast.

Figure 7(a) shows a low-magnification TEM bright-field image of a single belt with the width and length of about 800 nm and 1 μm, respectively. By comparing this with the SEM results, we suggest that the “region I” feature with a larger width may correspond to the bottom part of the heterojunction belt, while the sharp tip feature of “region III” may correspond to the top of the belt. The corresponding SAED pattern on the [0001] projection in Figure 7(a) shows that the entire belt is a single-crystal wurtzite structure growing along the [10-10] direction without any extra diffraction spots, indicative of epitaxial interfaces across the heterojunctions. However, the average indium concentrations with respect to ZnO acquired from EDS reveal large variations in spatial distribution, at about 10.3, 2.3, and 2.5 at. % for the bottom (I), middle (II) and top (III) regions of the belt, respectively. Notably, the nucleation region corresponds to the highest indium concentration.

To gain more insight into the microstructure and composition of the HB, the region marked with a white rectangle in Figure 7(a) is cut by FIB for TEM cross-sectional characterization. Figure 7(b) shows a bright field image in the upper part and STEM HAADF image in the lower part, which are analogous to the two SE images in Figure 6 with regard to contrast variation. Whereas the TEM bright field image indicates the smooth top and bottom surfaces as revealed by the SE image in Figure 6(d), the HAADF image demonstrates similar layer contrast to the SE image from the upper detector in Figure 6(e), in that the two side regions are brighter than the central part. The brighter regions are ascribed to a higher indium concentration, consistent with the EDS In map in Figure 6(g). The FIB-cut sample is further studied by TEM, EDS and SAED in Figure 7 (c) for image contrast, indium concentration, and diffraction pattern, respectively, by which three distinct regions (i, ii, and iii) are assigned. Region i (central part) is characterized by the single-crystalline wurtzite structure of ZnO with negligible indium concentration, as shown in the SAED pattern in the inset, and evidenced by the darkest contrast in the HAADF image and
the EDS characterization. In region ii, the average indium concentration is around 1.7 at%, which is associated with the brighter contrast in the HAADF image. The DP in the inset also corresponds to the single crystalline wurtzite structure, with the diffraction spots shifted to a slightly lower angle compared with that of region i, suggesting that this region is single crystalline ZnO homogeneously doped with In. Upon careful examination of the HAADF image, region iii is found to exhibit an amazingly complex structure composed of many indium-enriched layers, as evidenced by bright lines oriented parallel to the <11-20> direction, with a few lines extending slightly into region ii, causing weak superlattice spots and prominent streaks in the SEAD pattern. The average In concentration in this region is even higher than region ii of around 2.4 at%, as measured by EDS, which in combination with the superlattice results suggest that this region has a modulated homologous In2O3(ZnO)n structure. Notably, a slight increase in indium incorporation switches the homogeneous In doping in region ii to the superlattice structure in region iii.

Similarly, the In2O3(ZnO)n superlattice structure generally consists of atomic layers of InO2-octahedra separated by slabs of wurtzite InZn1nO(n+1)+ of varying thicknesses [1, 16], exactly as observed in the high-magnification HADDF image of region iii in Figure 7(d). The formation of a superlattice structure will induce a large strain between the ZnO slabs and InO2 insertion layers. The ends of the partial inclusions through incomplete indium lattice diffusion are thus usually associated with edge dislocations, as shown in the white dashed circle in Figure 7(d). The accumulative strain is relaxed via two routes. First, indium atoms distribute along the hexagonal c axis in zigzag modulated structures with the inclined angle of 53°, thus reducing total strain, as shown in Figure 7(e), preferably for thicker ZnO slabs, consistent with theoretical predictions [17]. In addition, the octahedral indium inclusion layers act as inversion domain boundaries (IDB) to relax the strain, as shown in Figure 7(f) in a high-resolution HAADF image, where the stacking sequence of the close-packed metal layers switches from ABAB to CACA passing the IDB. Figure 7(g) shows a projected line profile across an IDB, where the d-spacing in the InZn1nO(n+1)+ layer remains almost the same as in pure ZnO (0002) planes (0.26 nm), but with a 19 % increase in the d-spacing (0.31 nm) on the either side of the In-O insertion layer.

From the above analyses, we demonstrate that a novel 5-layer heterojunction ZnO belt comprised of In2O3(ZnO)n / In:ZnO / ZnO / In:ZnO / In2O3(ZnO)n is synthesized by a facile growth method. The possible growth mechanisms could be rationalized by four steps, as shown in Figure 7(h). (1) Self-catalyzed vapor-liquid-solid (VLS) combined with vapor-solid (VS) mechanism: In the beginning, alloy droplets are formed on the substrate from the co-condensation of zinc and indium vapors, followed by the nucleation of ZnO belts with supplied oxygen via a self-catalyzed VLS process. At the same time, indium is incorporated as a dopant. According to the calculations [18], the surface energy of ZnO (0001) will become smaller than that of the other major planes once indium doping exceeds 1/4. The fact that nucleation region I at the bottom is rich in indium makes the 1D ZnO grow in the form of a belt along the thermodynamically favored [01-10] direction rather than the more common [0002] one. Moreover, it is worth noting that the width is non-uniform throughout the belt, indicating that the
VS process is also operating[19], and that the decrease in width may be as a result of the varied growth rate in the [11-20] direction due to the gradual decrease in indium concentration. (2) Surface diffusion of indium: The indium concentration decreases from the bottom to top, implying that indium diffuses upward. Moreover, the large amount of indium available at the bottom establishes a large concentration gradient as the driving force for diffusion. Under this condition, surface diffusion is much more favored than slow lattice diffusion. (3) and (4) Redistribution of indium to form a heterojunction structure: Subsequently, indium diffuses inward to result in a three-zone sandwich-like belt structure, where each layer structure is determined by the average indium concentration and strain involved. Since the average indium concentration may exceed the solid solubility limit in ZnO in the outermost region (region iii), an In$_2$O$_3$(ZnO)$_m$ homologous structure is generated to compromise between indium concentration and strain. Region ii certainly results from residual diffusion of indium into ZnO lattices, a process that is limited by indium supply and temperature. The equilibrium structure is finally established through competition between belt growth and indium diffusion inward from the surfaces, causing region i top shrink more than regions ii and iii with regard to the width towards the top of the belt.

Figure 6. (a) Low-magnification SEM top-view image of the as-synthesized products on Si substructure. (b) and (c) high-magnification SEM images of nanodisks and microbelts, respectively. (d) and (e) SE images of a In$_2$O$_3$(ZnO)$_n$/In:ZnO/ZnO belt recorded using ET and TTL E x B detectors, respectively. The insets show the corresponding projected intensity line profiles. (f)-(i) FESEM images and the corresponding EDS element mapping results of In, Zn, and O, respectively.
Figure 7. (a) TEM bright-filed image of an In2O3(ZnO)n/In:ZnO/ZnO belt with indium concentrations in distinct regions (I, II, and III) obtained from EDS. The inset is the corresponding SAED pattern recorded from the entire belt structure. (b) TEM cross-sectional bright-field (up) and HAADF (down) images of an belt. (c) Medium-magnification HAADF image recorded from the rectangular area in (b) with SAED patterns and In EDS data obtained from regions i, ii, and iii. High-magnification HAADF images obtained from region iii in (c) showing (d) alternate stacking of In-O layers and In/Zn-O slabs along the c-axis, and (e) zigzag modulated structures in the In/Zn-O slabs. (f) Atomic-resolution HAADF image showing columns of Zn and In cations. (g) Intensity line profile and d-spacing across an IDB. (h) Schematic diagram of the proposed growth mechanisms of the belts.

4. **Even higher enhanced optical and thermoelectric properties of the novel heterojunction structure**

This novel heterostructure provides an ideal pathway to enhance electron conduction through the indium doped ZnO layer, and the homologous In2O3(ZnO)n layer contains numerous interfaces to impede phonon transportation. This structure can offer an efficient way of separating the transportation of electrons and phonons for high thermoelectric power factors in semiconductor devices.

The room-temperature CL spectrum of the belts in Figure 8(a) shows a strong ultraviolet (UV) emission centered at 385 nm, as well as a weak and broad green emission centered at 501 nm. In general, the CL emission of undoped ZnO is located at around 372 nm [20]. Therefore, the red shift of the UV emission at 385 nm of the belt may be ascribed to the narrowing of the band gap
due to doping,[19, 21, 22] as well as a heterojunction band alignment effect. In addition, the peak at 501 nm is attributed to the transition between the photoexcited holes and singly ionized oxygen vacancies of the ZnO structure [23]. Figure 8(b) shows a low-magnification SEM image of an belt, while Figure 8(c) is the corresponding monochromatic CL images for the emission at 385 nm. In spite of all the ZnO-based material, the results reveal an unusual phenomenon in non-uniform UV emissions over the entire structure, where the peak at 385 nm is mainly contributed by the two sided zones (In doped ZnO and In2O3(ZnO)n), and this provides further support for the doping-induced red shift. To better understand the reasons behind this abnormal luminescence phenomenon, the line-scan profiles of the indium concentration from EDS, energy-filtered SE imaging, and CL emissions at 385 nm across the same heterojunction belt, as shown in the inset, are compared in Figure 8(d). By referring to the STEM results shown in Figure 7(c), the profile can be divided into a five-layer sandwich-like symmetric structure with regions i, ii, and iii corresponding to ZnO, In:ZnO and In2O3(ZnO)n structures, respectively. The EDS line profile confirms the maximum indium concentration in region iii, which decreases gradually inward to region ii. On the other hand, the SE intensity profile exhibits dark contrast only in region ii, suggesting this region is doped with donors more heavily than the adjacent regions i and iii, leading to greater ionization energy for less SE signals [24, 25]. The luminescence at 385 nm is mainly emitted from region ii, and only partially from region iii. To gain a deeper understanding of this structure, a transistor device fabricated by e-beam lithography, as shown in Figure 9(a), is used to measure carrier concentration based on the field effect of IV characteristics. Accordingly, the carrier concentration is determined to be about 8.8 × 10¹⁹ cm⁻³, presumably the signature of the highly doped region ii, which is far above the semiconductor-metal transition of a Mott critical density of 4 × 10¹⁸ cm⁻³ for In doped ZnO [21]. Consequently, the energy bandgap would be narrowed [26, 27] in region ii to form a degenerate semiconductor, verifying the red shift in UV emissions. With these results, the band structure in contact across the five-layer heterojunction in a belt can be tentatively proposed in Figure 8(e). Considering band alignment with a constant Fermi energy level, region ii of highly doped In:ZnO closely resembles a quantum-well-like layer, having energy barriers with adjacent lightly doped ZnO and In2O3(ZnO)n layers. Here, we realize ZnO quantum-well-like structures in an all ZnO heterojunction belt, and these produce confined UV light emissions resembling those of a light-emitting-diode, as opposed to dark bands in the SE image, due to the higher ionization potential that occurs in a self-consistent manner. The possible radiative recombination routes are also indicated in Figure 8(e). Upon excitation, the electron-excited carriers will be driven to the quantum-well-like region ii for better confinement, and these subsequently recombine as route ① for the major emission. However, there is some uncertainty with regard to the band alignment for the valence band, which depends on the bandgap reduction in both regions ii and iii, resulting in emissions via recombination across interfaces into regions i and iii, like in route ② for a type II semiconductor, and this is also partially due to the band-tail states. However, this type of emission can only occur over a short distance across the interface, as seen for the interface between regions i and ii, and this process cannot account for the weaker emission in region iii.
Although the band offset should also exist for the interface between regions ii and region iii, the presence of both a large amount of defects and interfaces in region iii of the homologous structure might stop electrons from diving away and promote recombination through defect levels, as in route ③ depicted in Figure 8(e). Nevertheless, CL emissions are successfully confined in the two symmetric In:ZnO regions as quantum wells in this interesting heterojunction belt structure, which may have potential applications in optoelectronic devices.

Moreover, this unique belt is not only useful for optoelectronic applications, but is also an ideal structure for exploring thermoelectric effects, since the many interfaces introduced in region iii can impede phonon transportation with an electron conduction channel decoupled in region ii. To further explore the thermoelectric properties of the belt, in addition to the field-effect device shown in Figure 9(a), another device incorporating nano-heaters was also fabricated on a SiO₂/Si chip by e-beam lithography, as shown in Figure 9(b). The electrical conductivity and carrier concentration of the belt determined from Figure 9(a) are $4.48 \times 10^4 \text{Ω}^{-1}\text{m}^{-1}$ and $8.8 \times 10^{19} \text{cm}^{-3}$, respectively, while the Seebeck coefficient is determined to be $-63.32 \text{μV/K}$ from Figure 9(b), and the power factor can thus be calculated as $2.07 \times 10^{-4} \text{Wm}^{-1}\text{K}^{-2}$ at room temperature. Comparing these results with those of previous studies [1], the power factor of a single ZnO / In:ZnO / In₂O₃(ZnO)n heterojunction belt is larger by a factor of 20 than that of undoped ZnO nanowires (about $1.03 \times 10^{-3} \text{Wm}^{-1}\text{K}^{-2}$), and comparable to the best reported results for ZnO-based nanomaterials, including Sb-doped ZnO microbelts ($3.2 \times 10^{-4} \text{Wm}^{-1}\text{K}^{-2}$)[28] and IGZO homologous nanowires ($5-6 \times 10^{-4} \text{Wm}^{-1}\text{K}^{-2}$)[1]. The high power factor of the belt is attributed to the higher carrier concentration due to indium doping, as compared with that of about $3 \times 10^{17} \text{cm}^{-3}$ for undoped ZnO nanowires. The unique sandwich-like belt with a high power factor produced in this work may make possible more efficient thermoelectric device applications, because the good separation of the high-conductivity In:ZnO channels enables better carrier conduction and the layer-by-layer In₂O₃(ZnO)n structures can impede the propagation of phonons due to multiple scattering, as schematically shown in Figure 9(c) for a ZnO / In:ZnO / In₂O₃(ZnO)n belt. The homologous belt also acts as a double core-shell phonon transfer system to reduce thermal conductivity via the depression and localization of long wavelength phonons at the interfaces of the insertion layers in In₂O₃(ZnO)n regions, and the interfaces between ZnO, In:ZnO, and In₂O₃(ZnO)n regions [29]. Although the size of the belts is not yet optimized for use with thermoelectric devices, the ideal structure to separate electron conduction from phonon transportation can be easily achieved by one-step synthesis. The thermoelectric efficiency is expected to be further enhanced by reducing the widths of the In:ZnO and In₂O₃(ZnO)n regions in future work. Overall, this work demonstrates a concept to fabricate an efficient thermoelectric structure by generating indium doped ZnO channels for efficient carrier transportation and numerous interfaces to resist phonon propagation.
Figure 8. (a) Room-temperature CL spectrum, (b) SEM image, and (c) monochromatic CL image at 385 nm of a typical belt. (d) Line-scan profiles of indium concentration, energy filter SE image, and CL emission at 385 nm acquired from the rectangular area in the inset. (e) Schematic diagram of the proposed band structure and radiative recombination routes of the belt.

Figure 9. SEM micrographs of an belt-based (a) field effect transistor, and (b) thermoelectric device prepared by E-beam lithography. (c) Schematic diagram of the proposed mechanisms for electron and phonon transportation in an belt.

3-2 Piezoelectric Nanogenerators
1. **AC Nanogenerator based on GaN nanowires with 80 mV output voltage**

A single vertical integrated nanogenerator (VING) device based on GaN nanowires is successfully fabricated with output voltage of up to 80 mV. The role of carrier screening effect was first exploited in AC typed VING.

In the first part, piezoelectric nanogenerator (NG) performance is investigated with basic semiconductor material characteristics, in an attempt to push its physical limit by exploring its deep physical insight toward higher power NG. Except for geometrical design of nanowires in NGs, among the semiconductor characteristics, we choose to study the effect of carrier concentration, which is expected to be crucial for improving NG output performances by simulations. However, few reports have investigated this issue by experiments. Here, $n$-GaN nanowire (NW) arrays with a series of doping concentrations are assembled into vertical integrated nanogenerators (VING) with PMMA inserted into gaps between nanowires to increase mechanical robustness and prevent electrical shortening. The detailed VING device structure is sketched in Figure 10 (a), and the resulting peak output voltage and current density are about 80 mV and 10 nA/cm$^2$, respectively, demonstrated in Figure 10 (b) and (c). This NG output performance is Taiwan-leading in GaN-based piezoelectric NGs. Next, the excess carriers would screen the piezopotential, and degrade the output performance of a NG, but how strong it would be remains un-clear. Therefore, GaN NW arrays are prepared with doping concentration varying from $7.58 \times 10^{17} \text{ cm}^{-3}$ to $1.53 \times 10^{19} \text{ cm}^{-3}$, and the output performances are demonstrated in Figure 11 (a) to (d). By increasing the carrier concentration in the GaN NWs within the above range, the average output voltage drops from $\sim 70 \text{ mV}$ down to $\sim 4 \text{ mV}$, and the average output charge density, which is normalized from the current density for a fair comparison, decreases from $\sim 0.464 \text{ nC/cm}^2$ to $\sim 0.064 \text{ nC/cm}^2$. It is about 17.5-fold fall for output voltage when the carrier concentration increases by about 20 times. Strikingly, a good relationship is apparent between average output voltage drop and carrier concentration increment, indicating that the reduced output voltage is caused by the strong carrier screening effect on the piezopotential via increased electron density inside the GaN NWs. It is, for the first time, observed that the free carriers degrade the piezopotential output drastically by 17.5 times where the output piezopotential is almost diminished.

Subsequently, the maximum output power density is further investigated and can be calculated by the following equation: 

$$P_{\text{max}} = J_{\text{sc-max}} \times V_{\text{oc-max}}$$

where $J_{\text{sc-max}}$ and $V_{\text{oc-max}}$ refer to the maximum short circuit current density and open circuit voltage, and the calculated result is depicted in Figure 11 (e), showing monotonic decrease from $\sim 0.8$ to $\sim 0 \text{ nW/cm}^2$ with increasing carrier concentration. According to the results, the highest power output occurs at the lowest carrier concentration, while the power output vanishes at the highest carrier concentration simply resulted from the screened remnant piezopotential. Consequently, in AC typed NGs, the carrier concentration imposes a huge impact on the output characteristics and the lowest possible carrier concentration promises for producing a best power output and avoids the screening effect. This
conclusion is totally different from DC typed NGs, which require an optimum carrier concentration to generate a maximum electrical power. The systematic study of the dependence of the strong screening effect on the output of NGs is world-leading where a 20-fold drop in output voltage is observed, simply by increasing the carrier concentration.

![Diagram of VING device structure and electrical circuit schematic of a GaN NW array VING.](image)

Figure 10: (a) VING device structure and electrical circuit schematic of a GaN NW array VING. (b) and (c) output voltage and current density of a VING prototype with time, respectively.
Figure 11: (a) and (c) output voltage and current density of 4 VINGs with different carrier concentrations in n-GaN NW arrays; (b) and (d) extracted average output voltage and maximum charge density with carrier concentration. The inset in (d) is the current density versus carrier concentration and (e) power density of VINGs with respect to different carrier concentrations.

2. **Piezoelectric Nano-light-emitting diode (Piezo-Nano-LED) based on vertical ZnO nanowire arrays on p-GaN substrate by low temperature growth method.**

Piezoelectric ZnO nanowire arrays grown on a piezoelectric GaN thin film provides a perfect template for investigating both piezo-tronic and piezo-phototronic effects. The length and diameter of the as-grown ZnO nanowire arrays by a low temperature solution method can be readily controlled. With increasing the molar concentration of Zn precursors in the recipe, ZnO nanowire arrays can lengthen from 1.25 $\mu$m to 4.25 $\mu$m and widen from 250 nm to 750 nm, as depicted in Figure 12. The microstructure of the heterojunction device is studied by high-resolution TEM, shown in Figure 13. The ZnO nanowires are confirmed to be single crystalline hexagonal structure with free of planar or linear defects. Moreover, the diffraction patterns further indicate the epitaxial growth between the ZnO nanowires and the GaN thin film along the [0002] direction, implying the strong substrate guiding effect.

Toward the piezo-tronic and piezo-phototronic applications, the strain-induced current-voltage characteristics of the $n$-ZnO/$p$-GaN heterojunction device are preliminarily investigated, as demonstrated in Figure 14. Upon compressive straining, the forward current gradually decreases, but the reversed current increases. This phenomenon confirms that the...
piezoelectric charges interact with the depletion layer close to the heterojunction and the interfacial Schottky barrier between ZnO and metal electrode. The positive piezoelectric charges would lower the barrier height and the negative piezoelectric charges further raise the barrier, which illustrates the opposite current responses of the heterojunction device when subjected to compressive strain. As a consequence, the preliminary results manifest the effect of piezoelectric charges on the electronic device and open a new research field when the piezoelectric effect works with semiconductor and couples with photon.

Figure 12: Dependence of length and diameter of ZnO nanowire arrays grown on p-GaN thin film with molar concentration of Zn precursors. The Insets show the corresponding SEM images with respect to each molar concentration.
Figure 13: (a) Low magnification TEM image of ZnO nanowire arrays grown on a $p$-GaN thin film. (b) High resolution image of a single ZnO nanowire taken from the yellow region in (a). (c) Diffraction pattern of a single ZnO nanowire. (d) Diffraction pattern of the $p$-GaN thin film.

Figure 14: (a) Logarithm of current versus voltage plot of the heterojunction device on straining. (b) Extracted forward and reversed current at 20 V and -5 V biased voltage as a function of strain.

References

(Please highlight the results which are World-leading or Taiwan-leading.)
### IV. Contributions

(Please complete the table as below and describe the major contributions.)

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