Highly Conducting In$_2$O$_3$ Nanowire Network with Passivating ZrO$_2$ Thin Film for Solution-Processed Field Effect Transistors

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Nanowire field effect transistors (NWFETs) are of great interest for future nanoelectronic application such as logic circuits, chemical/biosensors, and memory/display devices. Among various nanowire materials, indium oxide (In$_2$O$_3$) has received much attention because of its chemical stability, high mobility, and low temperature processibility. 1D nanowires featuring unique crystalline structure and distinctive carrier transport characteristics enable outstanding electrical performance.\[^1\] However, when single nanowire is utilized for channel, its complicated growth and transfer processes are considered as an obstacle due to low yield and inconsistency in chemical and electrical properties. On the other hand, multiple nanowire networks have less deviation and enable more reliable device performance with higher yield, suggesting their great potential for electronic devices.

Another issue in nanowires is that the substantial number of oxygen-related surface states originating from the large surface to volume ratio results in high off-current ($I_{\text{off}}$), subthreshold swing (SS), turn-on voltage ($V_{\text{on}}$), and vulnerability to environmental stimuli. In this context, various studies have reported the passivation of oxide nanowires with organic or inorganic layers.\[^2\] However, such a passivation process requires selective deposition steps including an additional patterning process. The stacking two heterogeneous metal oxide layers, a process frequently used to engineer a channel layer in a thin film transistor, is an attractive method to passivate the surface of a channel. In this bilayer structure, a top semiconducting layer such as InGaZnO, InZnO, or HfInZnO confines a conducting bottom layer and resulting devices demonstrate high mobility and robust gate bias stability.\[^3\] However, this kind of confinement effect is valid only when the thickness of the bottom layer remains thin usually less than 5 nm; otherwise the channel becomes conductive and the device exhibits large negative $V_{\text{on}}$ and off-current ($I_{\text{off}}$).\[^4\]

In this study, we demonstrate solution-processed In$_2$O$_3$ NWFETs for conventional bottom gate top contacts (BGTC structure) with a multiple nanowire network. Electrospinning is utilized to readily produce a scalable continuous In$_2$O$_3$ nanowire network. After an annealing process to remove the polymer matrix, the polycrystalline nanowire network exhibits a conductive channel property. To improve the FET performance, a bilayer structure comprising insulating zirconium oxide (ZrO$_2$) and a nanowire network is adopted. Having a high binding energy with oxygen, ZrO$_2$ is favorable for the passivation of oxygen-related surface defects, firmly securing the nanowire network to the substrate. In addition, Lee et al.\[^5\] reported that electrons can pass through several insulating oxides when those oxides are combined with a semiconducting buffer layer. ZrO$_2$, specifically, has a low work function compared to those of other insulating oxides,\[^6\] so it is often applied to injection layer in organic light emitting diodes or resistive memory layers in resistive random access memory.\[^7\] With a ZrO$_2$ top layer, NWFET performance shows high current on/off ratio ($I_{\text{on}}/I_{\text{off}}$) and field-effect mobility ($\mu_{\text{FET}}$), and small SS and $V_{\text{on}}$. Apart from NWFETs, due to limitations of the analyses of nanowire/thin film structures, a bilayer with In$_2$O$_3$ thin film and ZrO$_2$ is fabricated to investigate the conduction mechanism, which is based on the energy band structure.

**Figure 1a** provides a schematic illustration of the fabrication process of the hybrid In$_2$O$_3$ nanowire/ZrO$_2$ thin film FET. Using a conventional single nozzle electrospinning of indium nitrate/PVP solution, indium/poly(vinylpyrrolidone) (PVP) composite fibers are obtained in the form of a fiber mat onto a p+ Si/SiO$_2$ (100 nm) substrate (**Figure 2a**). The substrate is used as gate/gate insulator. Subsequent thermal annealing on a hotplate at 400 °C results in an In$_2$O$_3$ nanowire network that adheres to the substrate (**Figure 2b** and **Figure S1, Supporting Information**). During thermal annealing, the decomposition of PVP reduces the size of the indium/PVP fiber and some of the fibers are welded spontaneously to form nodes and branches in the randomly oriented nanowire network. The ZrO$_2$ top layer is coated directly on the nanowire network by a solution-process using spin coating and annealing process. Finally, using e-beam evaporation, the source and drain electrodes are deposited on top of the In$_2$O$_3$ nanowire/ZrO$_2$ thin film composite (**Figure S1, Supporting Information**). The diameter of the indium oxide nanowires is a Gaussian distribution, in which the average size of 224 nanowires is 35.24 ± 4.80 nm (see Figure S3, Supporting Information). All peaks in the X-ray diffraction patterns in Figure 2c correspond to cubic In$_2$O$_3$ crystalline structure with...
primary (211), (222), (400), (440), and (622) planes at \( \theta = 21.4^\circ, 30.5^\circ, 35.3^\circ, 50.9^\circ, \) and 60.6°, according to the JCPDS (PDF#06-0416). Figure 2d shows a representative transmission electron microscopy (TEM) image of an individual nanowire. The In\(_2\)O\(_3\) nanowire is composed of polycrystalline nanocrystallites with a size distribution of \( \approx 10–15 \) nm. The interplanar spacings obtained from the diffraction pattern are 2.92, 2.52, 1.79, and 1.52 \( \AA \), representing cubic In\(_2\)O\(_3\) (222), (400), (440), and (622) planes, respectively. Figure 2f is a TEM cross-sectional image showing that a single In\(_2\)O\(_3\) nanowire is surrounded by a ZrO\(_2\) top layer.

Figure 3a shows transfer curves of hybrid In\(_2\)O\(_3\) nanowire/ZrO\(_2\) thin film FETs as a function of nanowire electrospinning time; detailed FET performance is summarized in Table 1. The device with 2 min of electrospinning time turns on at \( V_G = 12 \) V; as the electrospinning time increases and the channel coverage which is the ratio of the physical channel area covered by nanowires increases, the turn-on voltage shifts to the negative side. This phenomenon is caused by increasing carrier concentration in the channel area. Since each In\(_2\)O\(_3\) nanowire acts as a 1D conducting path connecting the source and the drain electrodes, and because each nanowire also contains carrier electrons, the increase in the channel coverage produces more paths for electrons resulting in a negative shift of the turn-on voltage and high mobility.\(^{[8]}\) The on-current increases with the electrospinning time and slightly decreases after 10 min. At high channel coverage, random overlap between nanowires can cause an increase in the number of carrier traps or scattering centers at the interface. Therefore, the contact resistance between the nanowires increases and the movements of the carrier electrons are disturbed, inhibiting any further increase in the on-current. In this regard, aligned nanowires could lead to superior FET performance with higher directionality and less scattering centers.\(^{[8]}\) Figure 3b shows the optimized hybrid In\(_2\)O\(_3\) nanowire/ZrO\(_2\) thin film FET; it is compared with the bare In\(_2\)O\(_3\) NWFET. The saturation mobility (\( \mu_{sat} \)) is obtained from the highest differential value of \( (I_D)^{1/2} \) versus \( V_G \) curve under the assumption that the composite channel is in the form of thin film.\(^{[9]}\) When the electrospinning time is 10 min, the saturation mobility (\( \mu_{sat} \)) is a thin film is 9.6 cm\(^2\) V\(^{-1}\) s\(^{-1}\), SS is 0.58 V per decade and \( I_{on}/I_{off} \) is \( \approx 10^7 \). On the other hand, the bare In\(_2\)O\(_3\) nanowire FET shows conductive channel performance with a high off-current of \( \approx 10^7 \) and high SS of 3.45 V per decade. Even though bare In\(_2\)O\(_3\) NWFETs show decreasing off-current with decreasing nanowire density, their high SS and low \( I_{on}/I_{off} \) are not suitable for FET application. Unpassivated oxygen-related defects generate excessive electron carriers resulting in high \( I_{off} \) and SS and degrade transistor performance.

Even when an insulating ZrO\(_2\) thin film is placed between the nanowire network and the top electrodes, the electrical current flowing through the nanowires is well-collected by the electrodes. There are technical difficulties in analyzing the chemical states and the electronic energy structure of the In\(_2\)O\(_3\) nanowires, which makes it hard to explain the current conduction through the ZrO\(_2\) thin film and the improvement in FET performance. Thus, for further investigation, the nanowire network is substituted with the In\(_2\)O\(_3\) thin film (Figure 1b). The In\(_2\)O\(_3\) bottom layer is coated via “aqueous route” and annealed at 400 °C.\(^{[10]}\) The thickness is varied by adjusting the concentration of the precursor solution. The TEM cross-sectional image indicates that the 0.2 m In\(_2\)O\(_3\) film is polycrystalline, containing
ZrO$_2$ film does not deteriorate the crystallinity of the bottom layer; its thickness is 6.5 nm (Figure 2g,h). Figure 3d shows the transfer characteristics of the In$_2$O$_3$ thin film/ZrO$_2$ thin film bilayer FET. The thickness of the In$_2$O$_3$ thin film is controlled by varying the concentration of the In$_2$O$_3$ precursor solution from 0.1 to 0.15 to 0.2 M. Single layer In$_2$O$_3$ thin film transistors (TFTs) show channel-thickness dependent behavior because the number of carrier electrons can be modulated by varying the thickness.$^{[11]}$ With a decrease in the concentration of the precursor solution, In$_2$O$_3$ TFTs exhibit lower off-current. However, the turn-on voltage remains at the negative side and the current on/off ratio remains high. Meanwhile, with the ZrO$_2$ thin film, the improvement of the TFT performance is more noticeable. The values of $V_{on}$ of the three devices are ~0 V and for the 0.1 M In$_2$O$_3$ TFT with the ZrO$_2$ film, $I_{on}$ decreases below 10$^{-11}$ A and SS becomes steeper. The effect of the ZrO$_2$ film becomes more obvious with a consideration of the output characteristics, as can be seen in Figure 3d. In$_2$O$_3$ TFTs with the ZrO$_2$ film show distinct decreases in current level. Because the single In$_2$O$_3$ TFT shows improved TFT performance with decreasing carrier concentration, it seems that the ZrO$_2$ film can affect the concentration of In$_2$O$_3$ films. Thus, the role of the entire ZrO$_2$ film itself can be seen to be on effective carrier suppressor rather than current blocking layer. Table 1 summarizes the TFT performance of 400 °C annealed In$_2$O$_3$ thin film/ZrO$_2$ thin film bilayer transistor.

Even though ZrO$_2$ thin film can act as a carrier suppressor, it is still difficult to understand how the channel electrons can be delivered to the top electrodes through the dielectric layer because this would increase the contact resistance between the In$_2$O$_3$ film and the top electrodes. In order to elucidate the underlying mechanism, the electronic structure is investigated. As a first step, the band gap ($E_g$) of each film is obtained from the optical transmittance (Figure S6, Supporting Information). The plot of $h\nu$ versus ($\alpha h\nu$)$^2$ was drawn as shown in Figure 4a,b by converting the wavelength into photon energy and calculating the absorption coefficient using Equations (1) and (2)

$$\alpha = -\frac{\ln T}{d}$$  

(1)

$$\alpha = \frac{A(h\nu - E_g)^{1/2}}{hv}$$  

(2)

where $\alpha$ is the absorption coefficient, $d$ is the thickness of the film, $A$ is a constant, and $hv$ is the photon energy. The optical bandgap can be determined by extrapolating the linear part to the abscissa; the intersection indicates the band gap of the film.
material. The band gap of In$_2$O$_3$ and ZrO$_2$ are at 3.4 and 5.3 eV, respectively.

A detailed energy band diagram can be established with information on the Fermi level and the valence band maximum. From the UP spectra (Figure 4c), the secondary electron region can be seen to be related to the Fermi level, and the intersection of the kinetic energy and the straight line fitted to the edge of spectrum refers to the energy from the vacuum level to the Fermi level ($E_{VL-F}$, work function). The high kinetic region on the right corresponds to the valence band maximum ($E_{VBM}$). The difference between the UV energy of He I (21.22 eV) and the highest kinetic energy corresponds to the energy between the Fermi level and $E_{VBM}$ ($E_{F} - E_{VBM}$). The work function of the In$_2$O$_3$ film is 4.86 eV and the ($E_{F} - E_{VBM}$) is $(21.22 – 18.08 = 3.14)$ eV. For the ZrO$_2$ film, the work function is 3.95 eV and the ($E_{F} - E_{VBM}$) is $(21.22 – 16.46 = 4.76)$ eV.

The difference between the conduction band minimum (CBM) and the Fermi level ($E_{F} - E_{CBM}$) is identical to the value of ($E_g - E_{VBM}$). Since In$_2$O$_3$ film annealed at 400 °C is a conductive n-type material, the Fermi level is close to the conduction band maximum and the $E_{F, CBM}$ is 0.26 eV. Even though ZrO$_2$ is generally regarded as an insulating material due to its large band gap, a natural tendency to form oxygen vacancies generates occupied defect states near CBM and ZrO$_2$ tends to be an n-type material as well.[6a,7b] Accordingly, the $E_{F, CBM}$ of the ZrO$_2$ film is 0.54 eV and the Fermi level is located in the upper half of the band gap. If we postulate an equilibrium after the contact of the In$_2$O$_3$ and ZrO$_2$ layers under a common vacuum level, the built-in potential between the two films would be $(4.86 – 3.95 = 0.91)$ eV and the conduction band offset (CBO, $\Phi_B$) would be $[(4.86 – 0.26) – (3.95 – 0.54) = 1.19]$ eV. Since the CBO is sufficiently large, the electron carriers in the In$_2$O$_3$ film find it difficult to pass through the ZrO$_2$ film (Figure 4e and Figure S8a, Supporting Information). However, for the ZrO$_2$ film deposited on the In$_2$O$_3$ film, as can be seen in Figure 4c,
the UP spectra shows a shift of the secondary electron cutoff to 4.70 eV from 3.95 eV, indicating that there is a vacuum level shift. This vacuum level modification can be achieved at the junction of two heterogeneous metal oxides. When the oxides have different oxygen densities, at the interface, oxygen atoms of the metal oxide with higher oxygen density become loosely bound and move toward the lower density side for structural stabilization. This process produces oxygen vacancy-interstitial pairs (denoted by a Frenkel type defect) along the interface and generates interfacial dipoles. The direction of the interfacial dipoles points toward the positively charged vacancies resulting in the lower vacuum level.

From the X-ray photoelectron spectroscopy (XPS) O 1s spectra (Figure S7, Supporting Information), it can be seen that the ZrO$_2$ film has a higher oxygen density than that of the In$_2$O$_3$ film. The structural stabilization achieved by the oxygen movement supplies oxygen atoms to the surface of the In$_2$O$_3$ film passivating its oxygen vacancies and accordingly reducing the carrier concentration (Figure 4d). At the same time, the interfacial dipole toward the ZrO$_2$ film leads to the downward VL shift. Since the VL is located 0.75 eV below the common VL before contact, both the built-in potential and the conduction band offset decrease to (0.91 − 0.75 = 0.16) eV and (1.19 − 0.75 = 0.44) eV, respectively (Figure S8a, Supporting Information). Due to the lowering of the conduction band offset, charge transport between the two layers is allowed and the electrical current flows from the In$_2$O$_3$ film to the source/drain electrode (Figure 4e).

Since the current conduction reflects the electrical contact at the junction, in order to confirm the conduction mechanism, the current through the In$_2$O$_3$/ZrO$_2$ bilayer is investigated. Figure 4f is a double-logarithmic J–E plot of the Al/In$_2$O$_3$/ZrO$_2$/Al device at various temperatures. The J–E curve at room temperature is a straight line of which the slope is 0.92; the current density gradually increases with increasing temperature. According to Equation (3)

$$J = \sigma E = q\mu E N_s \exp\left(-\frac{(E_c - E_f)}{kT}\right)$$

the slope, which is close to 1 in a log–log scale of the axes indicates that the electrical conduction is due to ohmic conduction, in which the ohmic contact at the junction should be
satisfied. Therefore, it is a reasonable explanation that the interfacial dipole lowers the energy barrier, so that electron carriers are injected into the conduction band. The ohmic contact remains independent of the ZrO₂ thickness and only the current density decreases due to the resistivity of ZrO₂ (Figure 4h). Figure 3f shows that as the thickness of ZrO₂ film increases, I_on decreases due to high resistivity. In contrast, I_off, V_on, and SS do not change because ZrO₂ film decreases the carrier of In₂O₃ by passivating the oxygen vacancies only at the surface and the reduced conduction band offset is retained. In addition, the current level increases with increasing temperature, which indicates the linear relation between the electrical conductivity and the inverse temperature. In Figure 4g, the Arrhenius plot (Equation (3)) is shown; the slope fitted by the conductivities was –4.42. As a result, the Fermi level of the ZrO₂ film is found to be about 0.87 eV below the conduction band minimum. This is a higher Fermi level compared with the value obtained from ultraviolet photoelectron spectroscopy (UPS) analysis but it is a higher Fermi level compared with the value obtained from operation.

CBO induced by interfacial dipoles is maintained during the operation of a gate field and because the concentration of the carriers that may be thermally generated is located is the upper half of the band gap. It has been reported that during the operation of bilayer TFETs, the gate bias bends only a small portion of the energy band very close to the gate insulator, whereas the whole energy band is modulated in single layer TFETs. Thus, the lowered CBO induced by interfacial dipoles is maintained during the operation.

Gate bias stabilities of In₂O₃/ZrO₂ bilayer FET are additional important findings (Figure S9, Supporting Information). Under positive gate bias stress (PBS) condition, the turn-on voltage shifts +5.0 V, whereas negative gate bias (NBS) condition causes no changes in the turn-on voltage. In terms of the PBS instability, because electrons captured by trap sites at the gate dielectric/channel interface deteriorate the effective formation of a gate field and because the concentration of the carrier in the active channel is sufficiently large to operate the device, a turn-on voltage shift is observed. However, under NBS condition, positive charges like oxygen vacancies and holes are trapped at the interface and degrade the stability. Because the overall density of oxygen atoms is high compared with that of the single layer In₂O₃ film and oxygen vacancies at the In₂O₃/ZrO₂ interface are held by ZrO₂ film, the NBS stability shows the improved result compared with previous reports on aqueous route metal oxide TFETs. Additional thermal stress results in a –1.2 V turn-on voltage shift and with illumination stress; the turn-on voltage shifts –6.3 V. Negative bias illumination thermal stress (NBITS) instability accompanies the increase in SS, indicating that charged defects are created. As light can generate e-h pairs in metal oxide material, NBITS instability normally causes serious damage to metal oxide TFETs. ZrO₂ has a larger band gap than those of conventional metal oxide semiconductors; also, the movement of oxygen vacancies is restricted inside the channel.

In conclusion, we have fabricated a hybrid In₂O₃ nanowire/ZrO₂ thin film FET. The FET performance of the In₂O₃ nanowire network exhibits conductive behavior; however, with a ZrO₂ top layer, the FET shows improved performance with $\mu_{sat} \approx 9.6 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$, SS of $\approx 0.58 \text{ V} \text{p}^{-1}$, and $I_{on}/I_{off}$ of $\approx 10^7$. The role of the ZrO₂ thin film is confirmed by investigation of the chemical states and the electronic energy band of the In₂O₃/ZrO₂ bilayer film, which includes (i) passivation of oxygen vacancies at the surface of the In₂O₃ film resulting in a decrease in the carrier concentration and (ii) generation of interfacial dipoles which enable electrons to be delivered to the top electrodes due to the lowered CBO. This mechanism is also compatible with the 1D In₂O₃ nanowires. Even though the average diameter of the In₂O₃ nanowires is thicker than the thickness of thin film, 1D nanowires have lower numbers of electrons than are present in the 2D film. The directionality of the nanowires is favorable for electrons to drift without scattering, and a large number of surface defects are effectively passivated leading to superior FET performance.

**Experimental Section**

Thin Film and Nanowire Transistor Fabrication: For the preparation of the indium oxide nanowire network, electrospinning was utilized. The electrospinning solution was 5 g of dimethylformamide (99.8%, Sigma-Aldrich) containing 0.5 g of indium nitrate and 0.7 g of PVP (M_w = 1 300 000 g mol⁻¹, Sigma-Aldrich). The solution was stirred overnight at room temperature. The electrospinning was performed with the synthesized solution at a feeding rate of 2 μL min⁻¹ using a syringe pump and at a constant DC voltage of 12 kV between the stainless steel foil, employed as a collector, and a syringe needle (25 gauge). The collected as-spun indium precursor/PVP composite fibers on the substrate were annealed on a 400 °C hotplate in air to obtain dense indium oxide nanowires. The precursor solution for indium oxide thin films was made by dissolving indium nitrate hydrate (In(NO₃)₃·xH₂O, 99.999%, Sigma-Aldrich) in deionized water. The zirconium oxide solution was made by dissolving 0.1 M of zirconium isopropoxide isopropanol complex (Zr(OCH(CH₃)₂)₄·(CH₃)₂CHOH, 99.9%, Sigma-Aldrich) in anhydrous 2-methoxyethanol (CH₃OC₂H₄OH, 99.8%, Sigma-Aldrich). The solution was filtered through a 0.2 μm membrane polytetrafluoroethylene (PTFE) syringe filter before spin-coating. The solutions were spin-coated at 5000 rpm for 30 s onto p-type Si substrate with a thermally grown 100 nm SiO₂ dielectric layer on top. The spun-on film was first placed on a 150 °C preheated hotplate for 30 s and directly transferred to a 400 °C hotplate in air and annealed for 1 h. For the measurement of the TFT performance, a 100 nm thick aluminum source and drain electrodes were deposited using an E-beam evaporator through a shadow mask under high vacuum around at 3 × 10⁻6 Torr. The deposited channel width and length were set at 1000 and 100 μm, respectively.

Film, Nanowire, and Device Characterization: The microstructures and morphologies of the films and nanowires were investigated using scanning electron microscopy (SEM, Hitachi S-4800) and TEM (JEOL JEM-2100F). In order to obtain the optical band gap of the In₂O₃ and ZrO₂ thin films, the transmittance of each film deposited on soda lime glass was measured by an ultraviolet–visible–near infrared spectrometer (UV–vis–NIR Spectrometer, UV 3101PC, Shimadzu). Light sources were a Deuterium lamp (190–300 nm) and a Halogen lamp (360–3200 nm) and the detectors were a PbS cell (NIR region) and a Pm-photomultiplier (VIS/UV region). In₂O₃ and ZrO₂ thin films and an In₂O₃/ZrO₂ bilayer film deposited on aluminum foil were analyzed with UPS (Sigma Probe, Thermo VG Scientifc). UV light (He I, 21.22 eV) was irradiated onto the surface of the film and photoelectrons are collected to the analyser, and the detectors were a PbS cell (NIR region) and a Pm-photomultiplier (VIS/UV region). In₂O₃ and ZrO₂ thin films and an In₂O₃/ZrO₂ bilayer film deposited on aluminum foil were analyzed with UPS (Sigma Probe, Thermo VG Scientifc). The data were collected using monochromatic Al K radiation (1486.6 eV) in an ultrahigh vacuum system with a base pressure of $\approx 10^{-10}$ Torr. As it was possible for the surfaces of the films to be contaminated by hydrocarbon compounds and adsorbed pollutants, the interiors of the films were
analyzed after Ar sputtering for 15 s. The Ar 2p peak at 241.9 eV was used for the calibration. The crystallinity of the In2O3 nanowires was confirmed using a High Resolution Powder X-ray diffractometer (HR Powder XRD, Rigaku). Nanowires after calcination were collected and attached to the holder by double-sided tape and Cu K-alpha radiation of 40 kV and 300 mA was used irradiate the sample. The scan step and speed were 0.01° and 4° min⁻¹. The electrical properties of the fabricated metal oxide TFTs were measured in the probe station with an HP 4156A precision semiconductor parameter analyzer. Output characteristics were measured as the voltage between the drain to source (VDS) increases from 0 to 40 V every 500 mV with the gate biases (VG) of −10, 0, 10, 20, 30, and 40 V. Transfer curves were measured as the gate bias was swept from −20 to 40 V at 500 mV intervals with VDS of 40 V. Output characteristics and transfer curves were plotted on I(D)–V(D) and log(I(D))–V(G) graphs.

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

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