

Electrospun Amorphous In-Zn-O Nanofibers: Nanostructural Engineering for High Performance Field Effect Transistors and Efficient Ethylene Gas Sensors

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1D oxide semiconductor nanofibers (NFs) have attracted significant interest as nanostructured electronic materials for universal electronic devices owing to their high surface-to-volume ratio, short charge transport pathways, and excellent mechanical flexibility and stretchability. In this study, electrospinning is used to prepare amorphous In–Zn–O (IZO) NFs, enabling the facile and direct fabrication of NF-based field-effect transistors (FETs). Compared with polycrystalline-phase counterpart, these devices exhibit superior electrical performance, with an electron mobility of $33.85 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, threshold voltage of -0.79 V , and a small subthreshold swing of 0.21 Vdec^{-1} at a low channel coverage of $0.35 \text{ } \mu\text{m}^{-1}$, facilitating low operating voltage. Furthermore, owing to the special electronic properties of amorphous oxide materials, characterized by fewer grain boundaries, and the high specific surface area of NF structures, the amorphous IZO-NF-based gas sensor exhibits exceptional sensitivity toward agricultural ethylene gas in a wide range of concentrations at low temperatures. These demonstrated versatile electronic applications of amorphous IZO NFs highlight their potential as candidate materials for future wearable electronics.

1. Introduction

In recent decades, metal oxide semiconductors (MOSs) have garnered considerable research interest worldwide owing to their

excellent optical-electrical properties, such as transparency, carrier mobility, stability, and uniformity.^[1–3] Enhancing thin-film transistor (TFT) device performance is crucial for enhancing display efficiency, and NF-structured metal oxides have been developed to achieve sustainable electronics. Specifically, the 1D nanostructure facilitates a shorter carrier transport pathway compared with that in conventional thin-film counterparts.^[4–6] Furthermore, the high surface-to-volume ratio provides numerous active sites, rendering NF-based oxides suitable for various electronic applications, such as gas sensors.^[7–9] Notably, MOS NFs demonstrate outstanding mechanical flexibility and stretchability.^[5,10–12] Therefore, FETs based on MOS NFs have emerged as an attractive candidate for future flexible displays.

Various experimental methods, such as hydrothermal^[13,14] and solid vapor

deposition^[15,16] techniques, have been used for synthesizing oxide semiconductor NFs. Among these methods, electrospinning is considered the most feasible and facile manufacturing technique for the direct synthesis of diverse functional NFs (organic, inorganic, and hybrid) for various applications, such as electronic devices based on semiconducting oxide NFs.^[12,17–19] Electrospinning enables the direct fabrication of these NFs with nanoscale diameters through precise control over the chemical composition of input materials (e.g., cation salts, solvent, and polymer matrix), operating parameters (to ensure the uniformity of NFs), and annealing conditions (to ensure the removal of organic components while forming a stable metal oxide network, thereby imparting electrical tunability).^[4]

Among the widely used MOS materials in TFTs, indium oxide (In_2O_3) is a conventional n-type semiconducting oxide known for its high carrier concentration resulting from abundant local oxygen vacancies.^[20,21] However, excessive oxygen vacancies can lead to electrical performance degradation and device instability, manifesting as high off current, large negatively shifted threshold voltage (V_{TH}), and low on/off current ratio ($I_{\text{ON}}/I_{\text{OFF}}$).^[21,22]

To address these limitations, IZO is considered a promising alternative owing to its excellent electrical characteristics and controllable carrier concentration, achievable by modulating the

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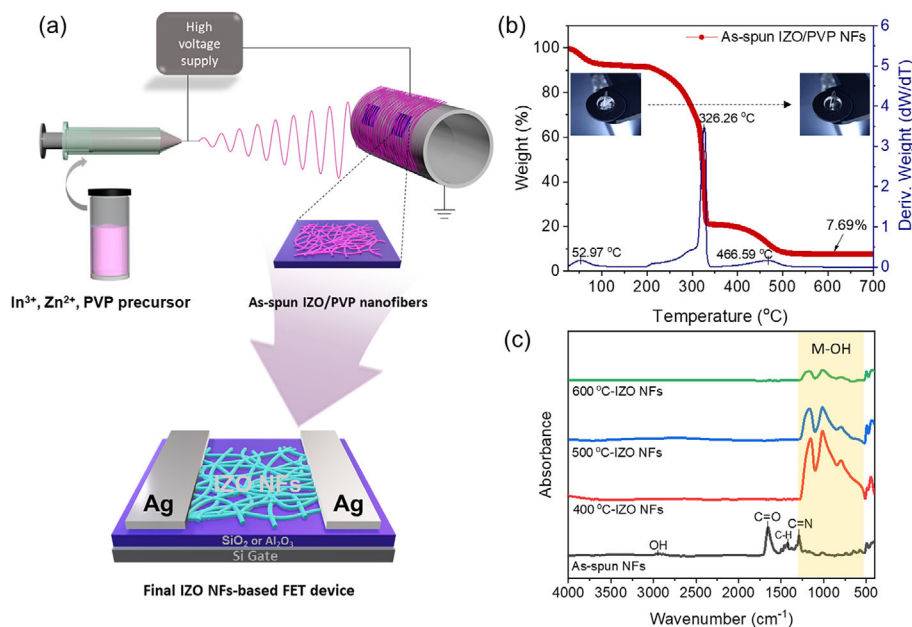


Figure 1. Fabrication and composition of IZO NFs. a) Schematic illustration of electrospinning of IZO NFs. b) TGA curves of IZO/PVP solution. c) FTIR spectra of as-spun and high-temperature-calcined IZO NFs.

stoichiometric molar ratio between In and Zn.^[23,24] In the IZO microstructure, the amorphous phase is typically dominant owing to the presence of Zn, which suppresses carrier generation by reducing oxygen vacancy formation, thereby enhancing electrical conductivity.^[25,26] Additionally, the amorphous structure, characterized by a disordered atomic arrangement and absence of grain boundaries, offers more uniform electron transfer across a large area while minimizing charge traps and electron scattering.^[27–29] Therefore, amorphous IZO in NF form, with its extremely high surface-to-volume ratio is expected to exhibit excellent sensitivity in gas-sensing applications.

There have been a numerous reports on the fabrication of IZO TFTs with relatively excellent electrical performance.^[30,31] Meanwhile, to our best knowledge, the experimental studies on IZO-NF-based FETs remain limited. Hence, in this study, 1D In–Zn–O NFs were fabricated via electrospinning, providing a comprehensive analysis of their microstructural transformation as a function of the atomic molar ratio of metal cations (In^{3+} and Zn^{2+}) and thermal calcination temperature. Moreover, their effects on the electrical performance FETs and ethylene gas-sensing performance of chemiresistive gas sensors were explored. This study unlocks the limitation on one of the presentative MOS in 1D nanofibrous structure and simultaneously offers a promising candidate for future electronic devices on both rigid and flexible substrates.

2. Results and Discussion

2.1. Microstructural Transformation of IZO NFs

2.1.1. IZO NF Fabrication and Material Characterization

Electrospinning of IZO nanofibers (Figure 1a) represents a facile and direct experimental method for fabricating NF-based thin

films with diverse material compositions. This process enables the flexible selection of precursor solutions, direct collection of NFs, and formation of 1D NFs. For IZO NFs, the optimal molar ratio between In and Zn was systematically investigated and shown in Figure S1 (Supporting Information) with the morphological, microstructural phase, and relevant electrical performance. It has been found that $\text{In}:\text{Zn} = 7:3$ (IZO73) is the optimal stoichiometric molar ratio for IZO NF fabrication. This molar ratio has been previously identified as corresponding to a high electrical performance in existing studies on IZO TFTs.^[32,33] The thermal decomposition behavior of the as-spun IZO73/PVP (PVP: polyvinylpyrrolidone) NFs was analyzed via thermogravimetric analysis (TGA). According to the TGA curves (Figure 1b), the weight reduced sharply at 326 °C, and the final decomposition peak appeared at 466 °C. Complete removal of the PVP polymer and organic compounds occurred at 500 °C, after which, the weight remained constant. To examine the presence and thermal decomposition of organic residues, Fourier-transform infrared (FTIR) spectroscopy was performed on as-spun and calcined IZO NFs (at 400, 500, and 600 °C). In the spectra (Figure 1c), the absorption peak near 3000 cm^{-1} was attributable to the stretching vibration of the hydroxyl –OH group formed via weak hydrogen bonding during electrospinning. Peaks at 1655, 1423, and 1290 cm^{-1} were ascribed to the vibrations of C=O, C–H, and C=N bonds of the PVP polymer, respectively.^[34,35] As expected, after high-temperature calcination, all these peaks belonging to organic residues nearly disappeared, indicating the removal of PVP. Additionally, three new adsorption peaks emerged at 1157, 1015, and 796 cm^{-1} , with their intensity reducing with increasing annealing temperature. The spectra at 400 °C exhibited the strongest peak intensity, likely because of the contributions of organic residues (Figure 1b), surface-adsorbed carbonate ($-\text{CO}_3$), and M–OH vibrational deformation bands.^[36–38] At higher calcination temperatures (500 °C), the peak intensity reduced owing

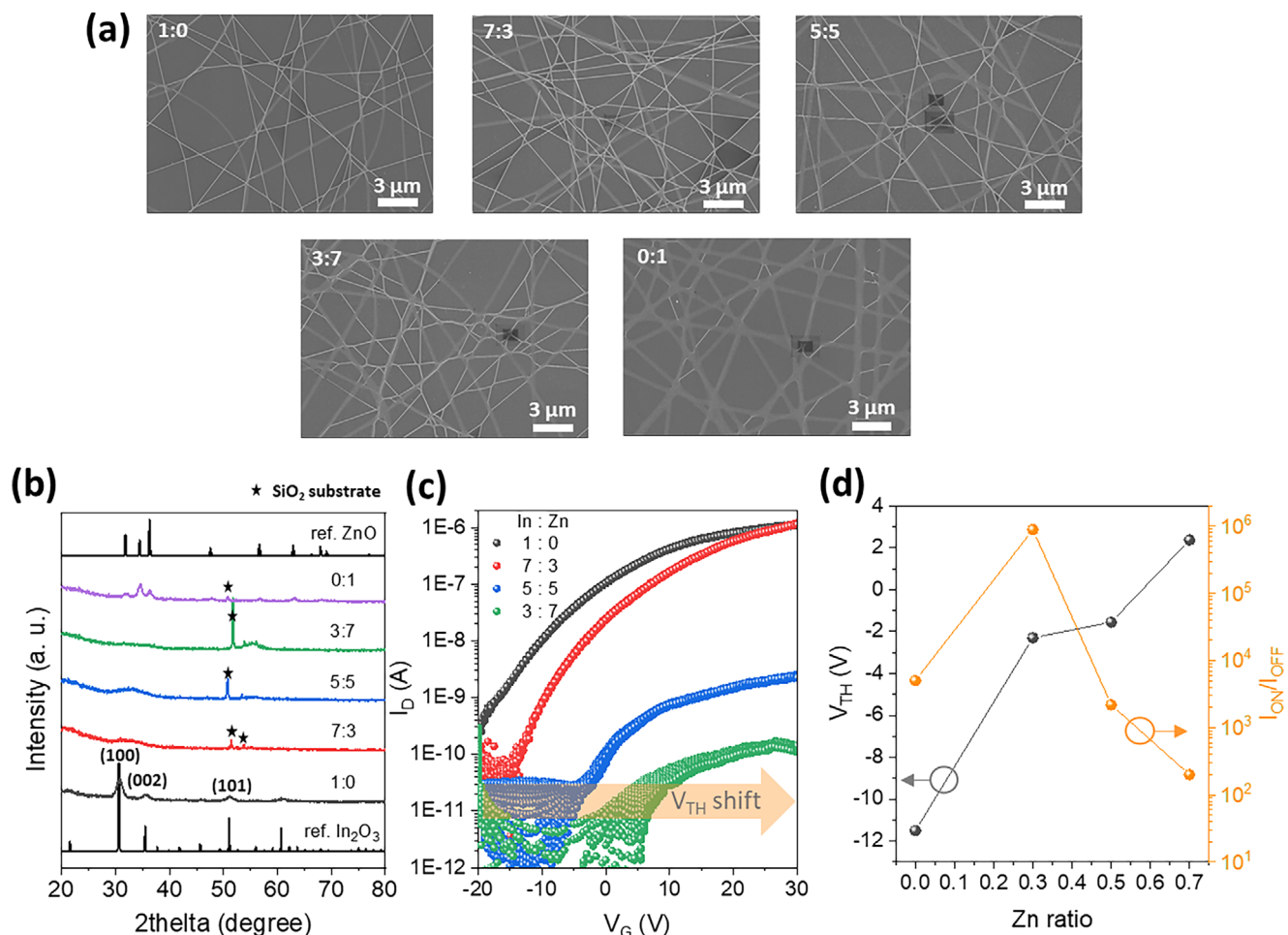


Figure 2. Effect of In:Zn molar ratio on morphology and electrical properties. a) FE-SEM images, b) XRD patterns, of IZO NFs, and c) transfer curves, d) relevant V_{TH} , I_{ON}/I_{OFF} , of IZO NFs-based FETs, with different molar ratios.

to the removal of residual organic species and improved structure order in the metal oxide network (In–O–In, Zn–O–Zn, and In–O–Zn), which diminished photo scattering. Therefore, subsequent analyses focused on the microstructural transformation and electrical performance of IZO NFs at temperatures exceeding 400 °C.

The morphology of IZO73 nanofibers was characterized by field-emission scanning electron microscopy (FE-SEM). A significant reduction in NF diameter was evident (Figure S1a,b, Supporting Information). Specifically, the average diameter of the as-spun IZO73 NFs (278.6 nm) decreased to $\approx$ 48.1 nm after the calcination at 500 °C for 2 h. This reduction was attributable to the removal of organic components and densification of the oxide structure through thermal hardening. Notably, welded junctions were observed between two adjacent IZO NFs after calcination, which is expected to enhance carrier transport and electrical performance.^[39] Figure S1c (Supporting Information) shows the STEM-energy dispersive X-ray (EDS) elemental mapping of a randomly selected IZO500 NF, indicating the uniform elemental distribution of In and Zn atoms in the NF shape, confirming the successful formation of IZO NFs.

2.1.2. Influence of In–Zn–O Molar Ratio

Figure 2 shows the influence of the stoichiometric molar ratio of In and Zn on the NF morphology and electrical performance of TFTs. Pristine In₂O₃ NFs (In:Zn = 1:0) exhibit a uniform shape (Figure 2a), with the XRD pattern revealing a typical crystalline phase with three characteristic XRD peaks of (100), (002), and (101) at 31.86°, 34.56°, and 36.35°, respectively (Figure 2b). Upon the incorporation of Zn into the indium oxide network, the fiber shape remained unchanged. However, the crystalline structure became disordered and transformed into an amorphous structure, as indicated by the disappearance of diffracted crystalline peaks and the emergence of broad XRD peaks. Similarly, pristine ZnO NF (In:Zn = 0:1) also exhibits a crystalline phase. In other words, the co-existence of binary oxides of In₂O₃ and ZnO increased atomic disorder in the microstructure, leading to amorphization of the lattice structure.^[40,41] FE-SEM images of IZO NFs with different molar ratios (Figure 2a) indicated that increasing the Zn content induced NF-melting behavior. At the same calcination conditions, the pristine ZnO NFs underwent complete melting, likely influencing the electrical performance (Figure 2c). The transfer curves of FETs based on IZO NFs at different

molar ratios (Figure 2c,d) demonstrate the dependence of electrical performance of NF-based FET devices on the molar ratio of In to Zn. The FET based on pristine In_2O_3 NFs exhibited a significantly negative threshold voltage ($V_{\text{TH}} = -11.5$ V), attributable to the presence of oxygen vacancies that generate excessive free carriers, a well-known characteristic of In_2O_3 .^[42] As the Zn content increased, V_{TH} gradually shifted to the positive direction, and the $I_{\text{ON}}/I_{\text{OFF}}$ value changed (Figure 2d). This variation in the electrical performance was likely associated with the incorporation of Zn atoms into the In_2O_3 structure, which altered the defect formation energy, especially for oxygen vacancies. In general, adjusting the molar ratio may help modulate vacancies and suppress excess carrier, thereby enhancing the electronic properties of In-Zn-O NFs.^[43] The optimal electrical performance was observed for the In:Zn = 7:3 composition, with an improved V_{TH} of -2.3 V and an $I_{\text{ON}}/I_{\text{OFF}}$ value of nearly 10^6 . Further increasing the Zn content led to deteriorated electrical performance, likely owing to reduced carrier concentration in Zn-rich IZO NFs. Additionally, the melted NF morphology likely limited the electrical performance, as it pertained to the evolution of the ZnO crystalline phase and formation of disconnected grains.

2.1.3. Effect of Calcination Temperature

The influence of calcination temperature on the morphology of IZO (7:3) nanofibers was microscopically investigated (Figure 3a). As the annealing temperature increased from 400 to 700 °C, the average NF diameter decreased, with the IZO700 NFs exhibiting an extremely small value of approximately 39 nm (Figure 3b). This reduction could be explained by the removal of organic components, as discussed, and the thermal densification of the metal oxide network, which contributed to a more compact nanostructure. Crystal grain growth became more apparent at temperatures exceeding 600 °C, and larger grains were observed at 700 °C, indicating a transformation from an amorphous phase to a crystalline phase. This transition was confirmed by XRD patterns (Figure 4a), which indicated a clear increase in crystallinity as the temperature rose. The sharp diffraction peak at 700 °C corresponded to the dominant crystallographic phase of cubic In_2O_3 . Figure 4b shows the electrical performance of FETs prepared using IZO NFs subjected to different calcination temperatures. The poor current observed in IZO400 NFs was attributable to the remaining organic residues, which functioned as charge traps. The IZO500 sample exhibited the optimal performance, and further increasing the temperature resulted in performance degradation. This deterioration was associated with the poly-crystallization of IZO, which led to the formation of numerous grain boundaries that resulted in electron scattering and hindered charge transport.^[44,45] Figure 4c presents the deconvoluted O 1s X-ray photoelectron spectroscopy (XPS) results, indicating the change in oxygen-related bonds, including lattice oxygen in the metal-oxide bond (M–O) at ≈ 529.2 eV, oxygen vacancies at ≈ 530.6 eV, and surface-adsorbed metal hydroxyl (–OH) at ≈ 532 eV. The concentrations of these components are shown in Figure 4d. The highest M–O bond concentration was observed in the IZO500 NFs, while higher calcination temperatures resulted in a decline in M–O bonds, likely

because of the generation of oxygen vacancies owing to M–O bond dissociation.^[46] Moreover, the reduction in M–OH bonds at higher temperatures is consistent with the FTIR results. The large number of oxygen vacancies at high temperatures likely contributed to the degradation of electrical performance observed in Figure 4b. Figure 4e illustrates the temperature-dependent microstructural phase transformation of IZO NFs, highlighting the transition from a disordered amorphous phase to a well-defined polycrystalline structure. Overall, the XRD and XPS results demonstrate the correlation between the NF microstructure and electrical properties, indicating the optimal calcination temperature for obtaining high-performance amorphous IZO NFs.

2.2. IZO-NF-Based FETs

2.2.1. Influence of NF Density on Electrical Performance

The electrical performance of IZO NFs FETs was investigated at different NF densities shown in Figure 5 and Figure S2 (Supporting Information). The channel coverage was determined based on the fiber density, calculated as the total length of NFs in a specific area (Figure S2a,b, Supporting Information). The effective channel width was determined by multiplying the number of oxide NFs in the overall TFT channel by their average diameter (Figure S2c, Supporting Information).^[17] The effective saturation mobility was then calculated based on the effective width. Figure 5a shows that as the NF density increased, the drain current increased, and a negative shift in V_{TH} was relatively observed. This could be attributed to the excessive carrier concentration associated with increased channel coverage. It is worth noting that the representative transfer curves in Figure 5a were selected from a larger set of devices, shown in Figure S3 (Supporting Information), measured at each distinct NF density. A slight variation was observed on each condition probably due to the inherent randomness in electrospinning process. Figure 5b and Figure S4 (Supporting Information) show key electrical performance parameters, including saturation mobility (μ_{sat}), $I_{\text{ON}}/I_{\text{OFF}}$, V_{TH} , and subthreshold swing (SS), along with their corresponding standard deviation. As the fiber density increased, μ_{sat} gradually increased from $2.95 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ at $0.35 \mu\text{m}^{-1}$ to $22.46 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ at $1.45 \mu\text{m}^{-1}$. In contrast, the $I_{\text{ON}}/I_{\text{OFF}}$ value reduced from 10^4 to 10^2 , indicating the inferior switching behavior and unreliability of FETs at high NF density. Moreover, V_{TH} shifted from -2.97 V at the lowest channel coverage to ≈ -10.06 V at the highest channel coverage. The gradual rise in SS confirmed the deterioration in device performance of IZO-NF-based FET as the NF density increased. Although the device with high NF density exhibited a superior mobility, the inherent inverse correlation with switching ability and SS imposes limitations on its practical implementation. Based on these findings, an NF density of $\approx 0.3\text{--}0.4 \mu\text{m}^{-1}$ may be optimal for FET applications. The electrical performance variation of IZO500 NF-based FETs at the NF density of $0.35 \mu\text{m}^{-1}$ is presented in Figure S5 (Supporting Information), showing 10 different transfer curves to illustrate the electrical performance uniformity of IZO NF-based FETs. This indicates the inferior uniformity

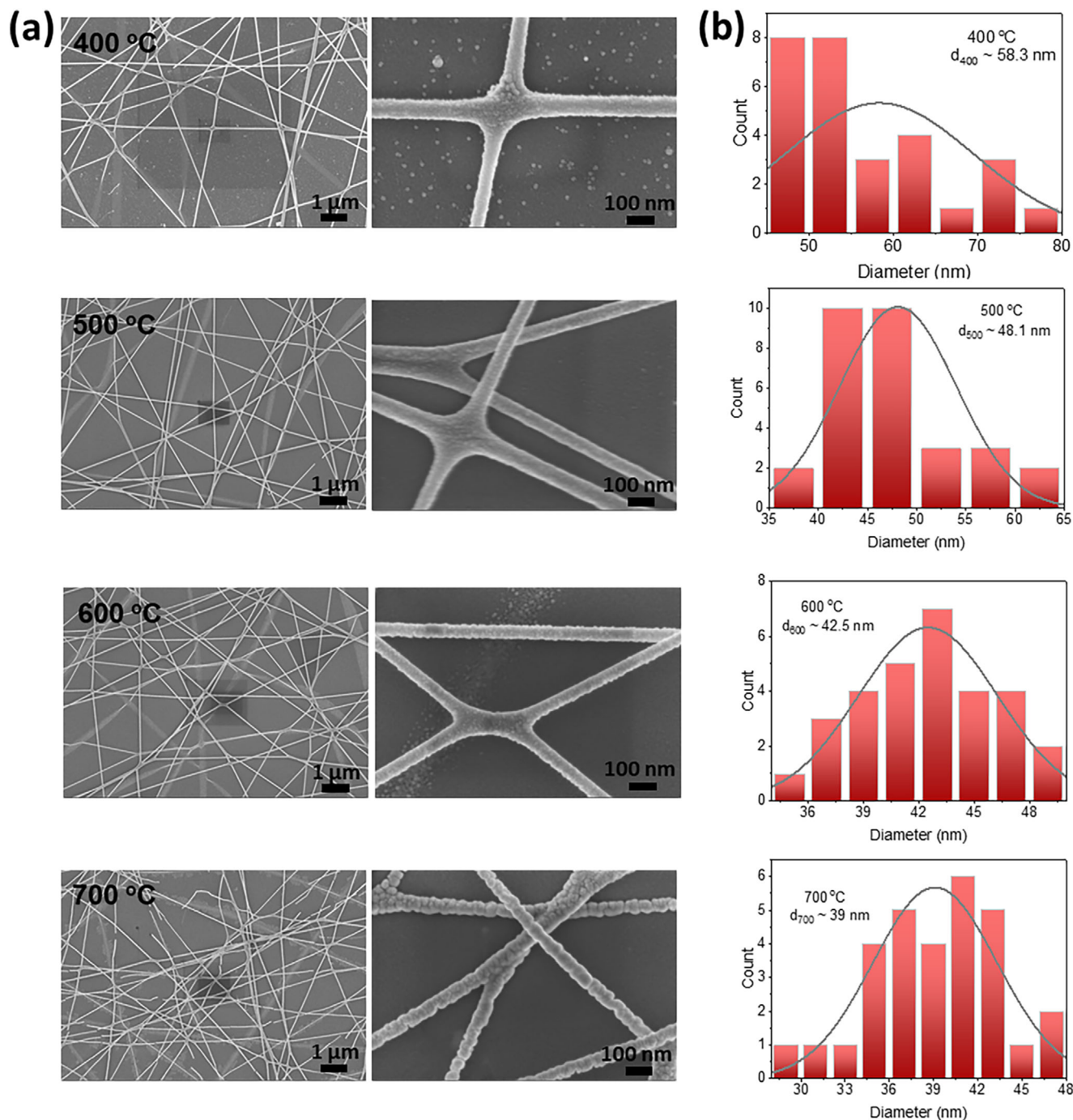


Figure 3. Morphology of IZO (7:3) NFs at different annealing temperatures. a) FE-SEM images. b) Histograms of average fiber diameter.

of randomly oriented nanofiber-based FET devices compared to the reported aligned-nanofiber-based FETs.^[17,18] Figure 5c,d shows the transfer and output curves for the most optimal device (relative NF density of 0.35 μm⁻¹). This device, with a low channel coverage, exhibited a reasonable transfer curve with μ_{sat} of ≈3.6 cm²V⁻¹s⁻¹. The output characteristics in Figure 5d indicated typical n-channel behavior with clear linear and saturation regions, indicating the operational stability of this IZO-NF-based FET.

2.2.2. Fabrication of IZO NFs on High-k Dielectric Al₂O₃

To enhance performance and reduce power consumption, IZO NFs were fabricated on a solution-processed Al₂O₃ gate dielectric. The structure of the resulting FET is shown in Figure 6a. The cross-sectional FE-SEM image in Figure 6b shows that the Al₂O₃ thickness was ≈89 nm. Moreover, strong adhesion between the IZO NFs and bottom Al₂O₃ dielectric was observed, indicating excellent interfacial contact between the NF channel and

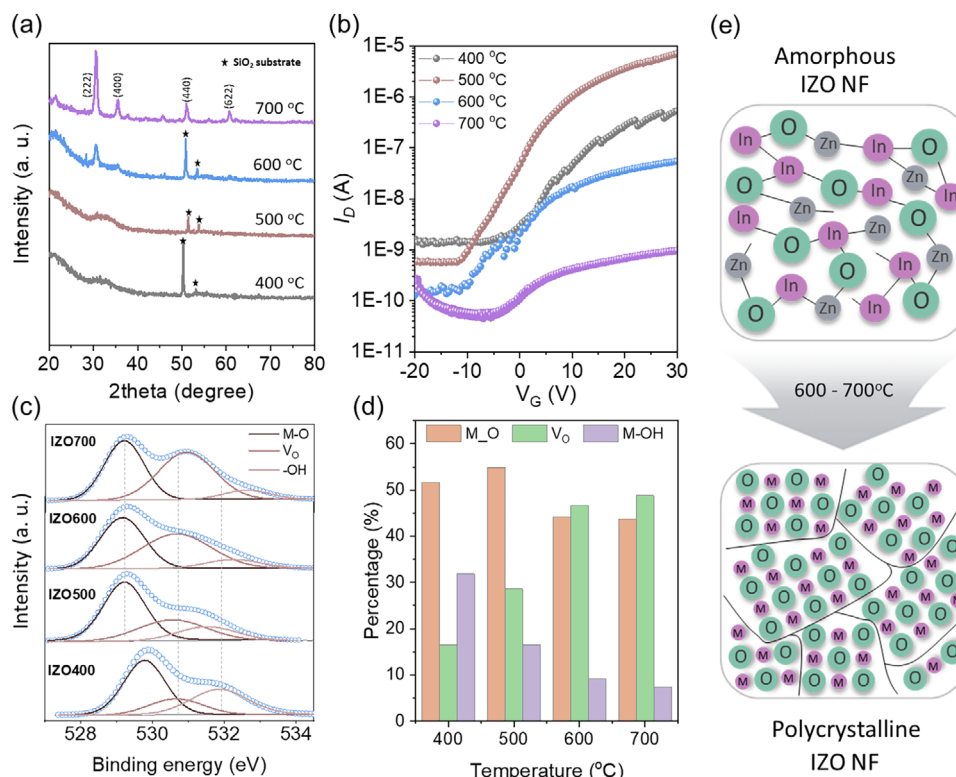


Figure 4. Effect of calcination temperature on IZO (7:3) NF properties. a) XRD patterns; b) transfer characteristics; c) deconvoluted XPS O 1s peaks; d) relative percentage of deconvoluted O 1s XPS peaks of IZO NFs at elevated temperatures, and e) illustration of metal oxide network phase transformation.

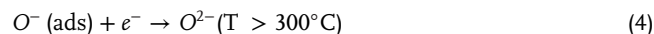
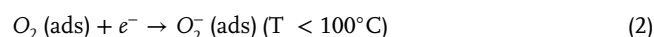
insulating layer. The dielectric properties of Al_2O_3 were measured using an LCR meter (ZM2376, NF Corporation). As shown in Figure 6c, the Al_2O_3 gate dielectric exhibited an areal capacitance C_i of 53.58 nF cm^{-2} and equivalent dielectric constant k of 5.58 at 1000 Hz. The leakage current profile of the Al_2O_3 insulator, shown in Figure 6d, reveals that the solution-processed Al_2O_3 could sustain a high breakdown electric field of up to 6 MV cm^{-1} . Compared with a commercial SiO_2 substrate, the solution-processed Al_2O_3 enabled FET operation at a voltage nearly 7.5 times lower. Thus, the electrical transfer curve in Figure 6e displayed improved performance, with μ_{sat} reaching $\approx 33.85 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, nearly 10-fold that of the SiO_2 -based FET device. Moreover, the $I_{\text{ON}}/I_{\text{OFF}}$ of the fabricated device exceeded 10^5 , indicating excellent switching function. V_{TH} of the IZO-NF-based FET was $\approx -0.79 \text{ V}$, and the SS was significantly reduced to 0.21 V dec^{-1} . Furthermore, the pinch-off behavior in the output curve shown in Figure 6f indicated the improved performance and reliability when using solution-processed Al_2O_3 as the gate dielectric for IZO-NF-based FETs at low operating voltages.

2.3. IZO-NF-Based Ethylene Gas Sensors

To further explore the electrical tunability of amorphous and polycrystalline IZO NFs at different temperatures and expand their potential applications, gas sensors based on 1D IZO NFs were fabricated for detecting agricultural ethylene gas. Figure 7a

depicts the chemiresistive gas sensor structure, where IZO NFs were directly deposited onto an indium tin oxide (ITO) interdigitated electrode (IDE) via photolithography. An optical microscope image of the sensor device revealed a dense IZO NF network. When exposed to reducing ethylene gas, ethylene molecules are adsorbed onto the IZO surface. Notably, the IZO with 1D NF shape exhibited a high surface-to-volume ratio, which could enhance target gas adsorption and desorption.^[47–49]

Research has shown that when oxide surfaces are exposed to air, oxygen molecules adsorb onto the surface and capture electrons from the conduction band, forming oxygen ions (O_2^- , O^- , and O^{2-}) on the surface of IZO NFs.^[50,51] Depending on the operating temperature, surface-adsorbed oxygen ions exist in different states.^[52]



The IZO NF surface is then considered to be oxidized; in other words, an electron depletion layer is formed on the surface, increasing the resistance. Upon exposure to ethylene gas, the ethylene molecules react with the adsorbed oxygen ions,

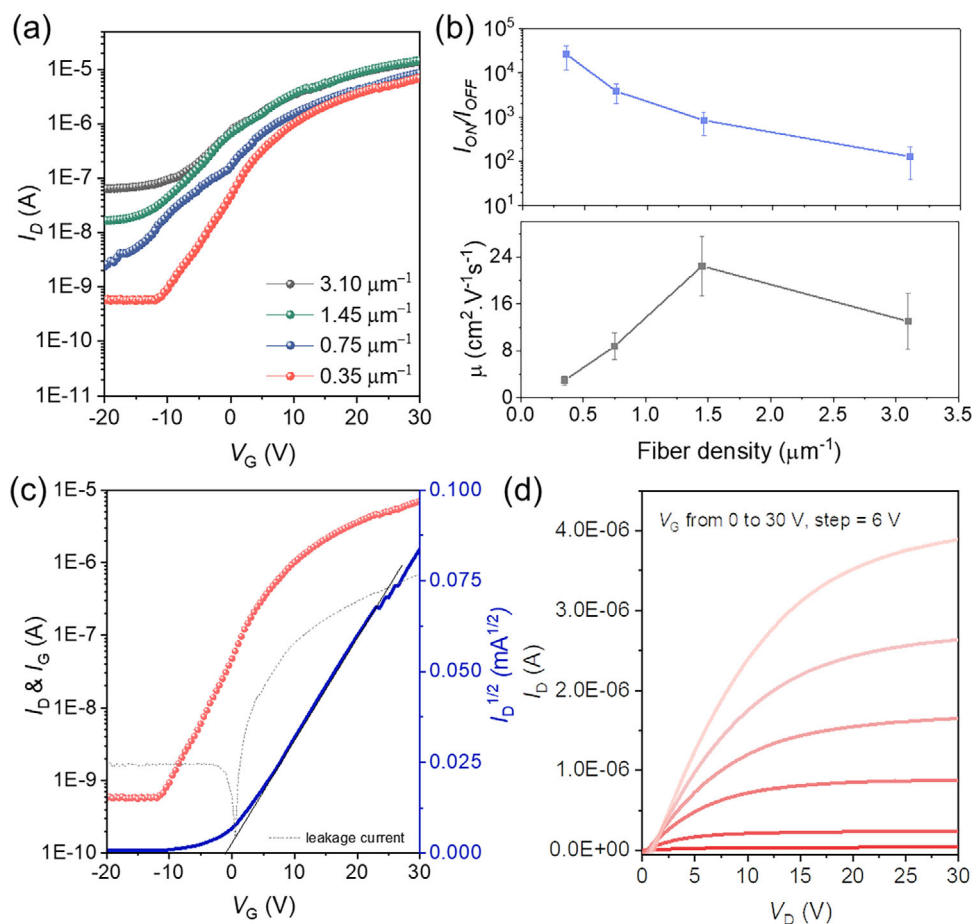
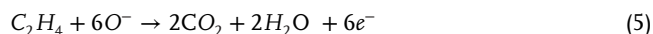


Figure 5. Electrical performances of IZO NFs based TFTs. a) Transfer curves, and b) Electron mobility and I_{ON}/I_{OFF} , of IZONFs-based TFT with various nanofiber density. c) Transfer and d) output characteristics of the optimal IZONFs-based TFT.

producing CO_2 and H_2O , while simultaneously releasing electrons back into the core conducting NFs. Consequently, the electron depletion layer becomes thinner, and the resistance of IZO NFs increases.^[53]



To determine the optimal operating temperature for the IZO NF-based gas sensor, the IZO500 NF sample was tested at elevated temperatures ranging from 150 to 300 °C. As shown in Figure S6 (Supporting Information), the IZO500 NF gas sensor exhibited the highest response $\approx 81.86\%$ (1000 ppm) at 200 °C, indicating this temperature as the optimal operating condition for the sensor. Figure 7b shows the sensing response of IZO NFs calcined at different temperatures when exposed to 200 ppm of ethylene at an operating temperature of 200 °C. The IZO400 and IZO500 devices, characterized by amorphous structures, exhibited sharp responses of 51.1% and 69.38%, respectively. The IZO400 device exhibited a severe signal drift when exposed to ethylene. This phenomenon could be attributed to the organic residues preventing the complete conversion of ethylene gas on the surface of the NFs. The IZO600 device showed no response to ethylene, likely owing to the lower concentration of chemisorbed oxygen ions (as indicated in the XPS spectra in

Figure 4c) and the formation of grain boundaries, which hindered electron transport.^[28,54,55] In contrast, the IZO500 sensor, with insignificant organic residues and an adequate amount of chemisorbed oxygen ions, afforded a suitable environment for ethylene gas conversion. Figure 7c,d show the dependence of sensing response on the ethylene gas concentration. The IZO500 NF sensor exhibited a linear correlation over a wide range of ethylene concentrations. Notably, three different sensors were fabricated under the same experimental conditions and measured for a better observation of the sensing performance uniformity. As shown in Figure S7 (Supporting Information), the three IZO500 NF-based sensors exhibit nearly similar linear and saturation response regions at elevated ethylene concentrations. This result indicates the high sensitivity and reliability of the proposed sensor device based on IZO NFs. To demonstrate the reliability and stability of the sensor, Figure 7e presents the results of five consecutive sensing cycles. A steady response was observed, with fast response and recovery times of 20 s and 176 s, respectively. Overall, the sensor device based on IZO500 NFs exhibited excellent sensitivity toward ethylene. This enhanced performance can be attributed to several factors. First, the nanofibrous structure with a small diameter offers a high surface-to-volume ratio, which, combined with its amorphous nature, facilitates efficient electron transfer.

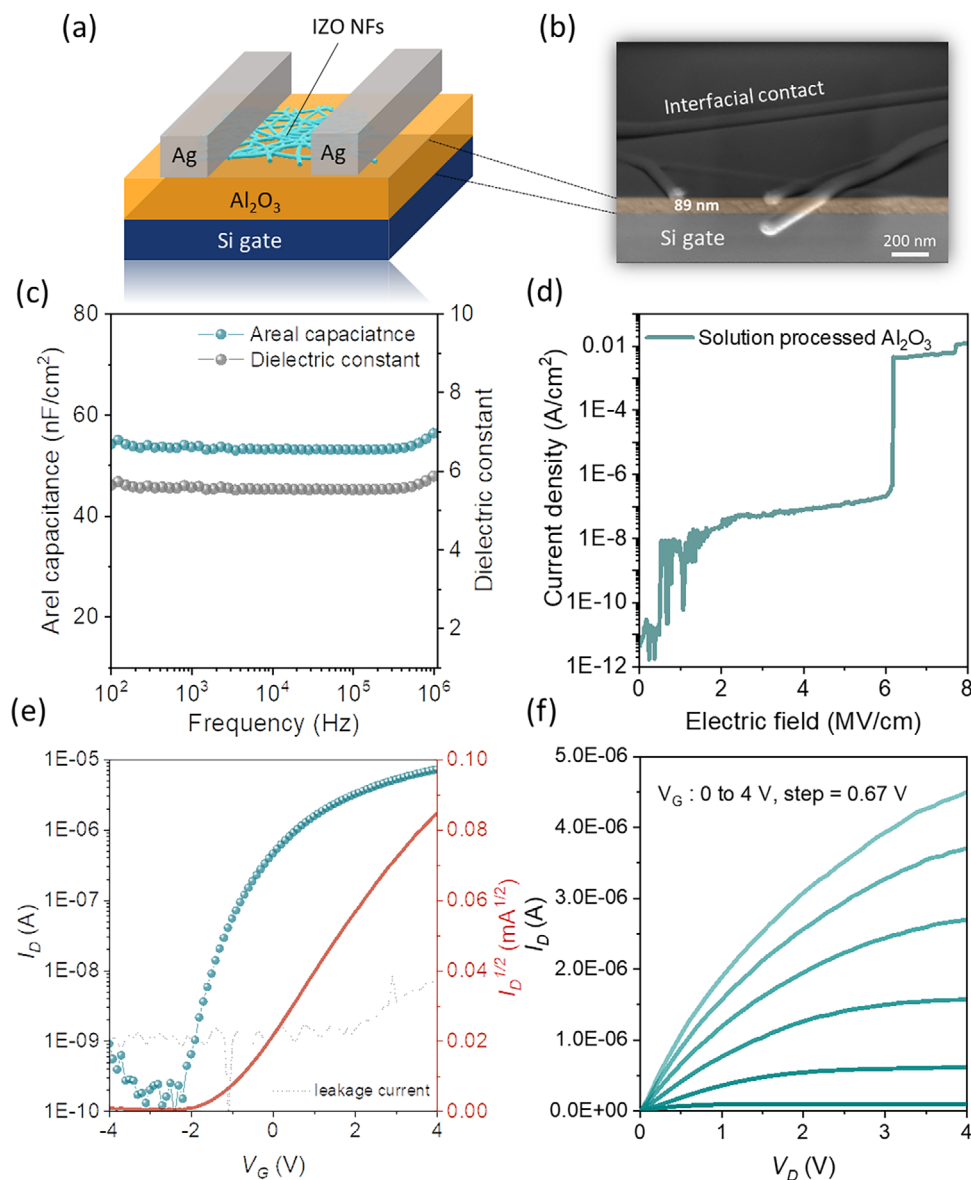


Figure 6. Electrical performance of IZO NFs on solution-processed Al_2O_3 . a) Illustration of IZO NFs/ Al_2O_3 based FET device structure; b) Cross-section SEM image of IZO NFs/ Al_2O_3 ; c) Areal capacitance and d) Leakage current profile, of Al_2O_3 dielectric; e) Transfer curve and b) Output curve of IZO NFs/ Al_2O_3 FET.

Second, the formation of an n-n heterojunction between the two n-type oxides (In_2O_3 and ZnO) contributes to the formation of a depletion layer at the interface due to electron transfer from In_2O_3 to ZnO , as the Fermi level of In_2O_3 is higher than that of ZnO ($E_{F(\text{In}_2\text{O}_3)} > E_{F(\text{ZnO})}$).^[56]

Moreover, the Pt coating significantly contributed to the sensing performance of the IZO NF-based sensor due to its catalytic properties. The decoration of Pt on the oxide surface provides additional active sites for oxygen adsorption. Furthermore, Pt-decorated IZO NF facilitates the spillover effect during oxygen adsorption, wherein oxygen species migrate onto the IZO NF surface, resulting in a higher concentration of chemisorbed oxygen ions available for reaction with ethylene gas, as described in Figure S8a (Supporting Information).^[57]

In addition, Pt possesses a relatively high work function (5.65–6.1 eV) compared to IZO, which promotes electron transfer from IZO to Pt. This charge transfer leads to the expansion of the electron depletion layer at the IZO NF surface, thereby enhancing its sensitivity to ethylene gas.^[58] Figure S8b (Supporting Information) presents the sensing response of the IZO500 NF sample with and without Pt coating. Notably, the sensor exhibited no measurable response in the absence of Pt, emphasizing the crucial role of Pt in the ethylene gas sensing mechanism.^[59,60] Future investigations may focus on further elucidating additional sensing characteristics of IZO nanofiber-based ethylene sensors, with particular emphasis on enhancing selectivity and optimizing performance at low operating temperatures.

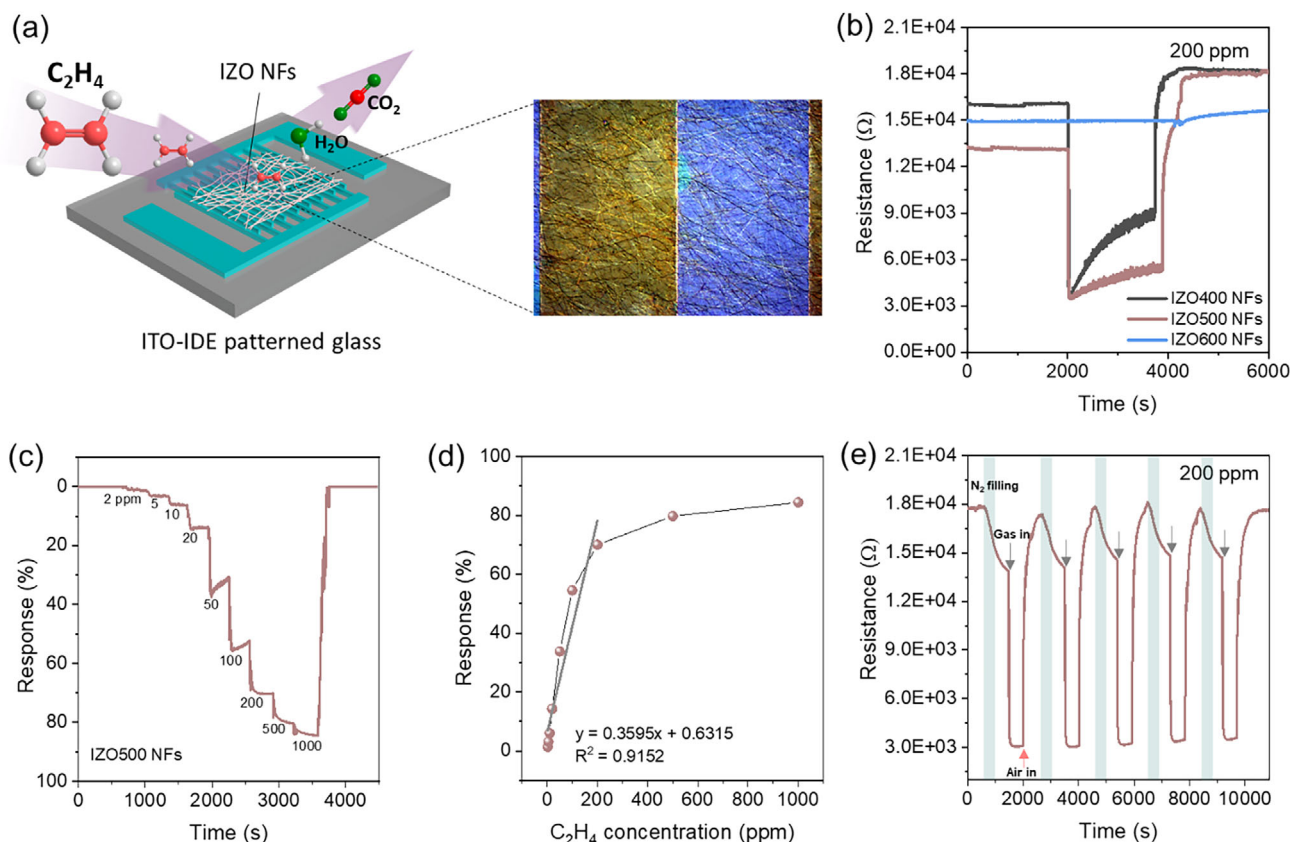


Figure 7. Ethylene gas sensing performance of IZO500 NFs. a) Schematic of IZO-NF-based sensor, chemical conversion of ethylene gas, and optical image of a part of the sensor; b) Sensing response of IZO NFs calcined at various temperatures toward 200 ppm of ethylene at 200 °C; c–d) ethylene gas concentration dependence of sensing response of IZO500 NFs; e) Five consecutive sensing cycles of IZO500-NF-based-sensor at 200 °C.

3. Conclusion

In conclusion, the atomic microstructure of IZO NFs was investigated. The results demonstrated the excellent electrical performance and gas sensitivity of amorphous IZO NFs when integrated in FETs and ethylene gas sensors, respectively. The resulting FET exhibited a high electron mobility of $33.85 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ while maintaining a low power consumption. Furthermore, when subjected to 200 ppm of reducing ethylene gas, the amorphous-phase IZO NFs, characterized by effective electron transfer, displayed a high response of 69.38% within 20 s at 200 °C. The demonstrated multifunctionality of electrospun amorphous IZO NFs can provide guidance for developing advanced 1D semiconductor metal oxides for future electronics.

4. Experimental Section

Polymer Solution Preparation: Indium nitrate hydrate ($\text{In}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$, 99.9%), zinc nitrate hydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 5\text{H}_2\text{O}$, 99.9%), and PVP (MW = 1300 000 g mol^{-1}) were purchased from Merck (South Korea); and dimethylformamide (DMF, 99.5%) was purchased from TCI. All materials and chemicals were used as received without further purification. $\text{In}(\text{NO}_3)_3$ was stoichiometrically mixed with $\text{Zn}(\text{NO}_3)_2$ at molar ratios of 1:0, 7:3, 5:5, 3:7, 0:1 in 5 mL of DMF. The total molar concentration was 0.2 M. The solution was stirred for 1 h, followed by the

addition of 1.2 g of PVP. The polymer solution was vigorously stirred for 12 h until a transparent and viscous polymer solution was obtained.

Electrospinning of In–Zn–O Nanofibers and FET Device Fabrication: The prepared precursor solution was loaded into a syringe and placed on the movable supporting frame of an electrospinning system (ESR200RD, NanoNC Co. Ltd., South Korea). The metallic spinneret nozzle (25G) was connected to the syringe. The polymer solution was electrospun at a relative flow rate of 1 mL/h and high supply voltage of 15 kV. NF jets were collected on a rotating drum collector (300 rpm) covered with aluminum foil. The SiO_2/Si substrate, cleaned using acetone and isopropyl alcohol, was directly affixed to the drum collector for collecting the NFs subjected to electrospinning for different durations at varying NF densities. The IZO/PVP NFs were successively calcined in a furnace (SH-Scientific) at temperatures of 400, 500, 600, and 700 °C for 2 h and denoted as IZO400, IZO600, and IZO700, respectively. The process was carried out under a humidity level below 30%. The solution-processed Al_2O_3 insulating layer was spin-coated onto a bare Si substrate, referring to an existing approach.^[61] The dielectric properties of Al_2O_3 dielectric were reverified. Lastly, 100-nm-thick Ag source and drain electrodes were thermally deposited using a shadow mask, forming a channel with a width and length of 1000 and 50 μm , respectively. The effective width was determined based on the NF density.

IZO-NF-Based Gas Sensor Fabrication: A chemiresistive-type gas sensor structure was chosen for IZO NFs considering its high simplicity and robust sensitivity. The ITO glass substrate was cleaned and patterned using photolithography to create IDEs with a spacing of 100 μm . IZO NFs were directly electrospun onto the IDE-patterned ITO substrate using the same polymer solution, followed by calcination in the furnace at various temperatures for 2 h. Next, Pt, as a catalyst, was sputtered onto the

calcined IZO NFs for 20 s. Prior to gas-sensing measurements, the gas sensor devices were soft-baked at 180 °C for 1 h to eliminate moisture.

Precursor and Nanofiber Characterization: TGA of the as-spun NFs was performed using a TGA550 analyzer (TA Instruments). FTIR analysis was performed using the FT/IR-8X instrument (Jasco Inc., Japan). Nanofiber morphologies were assessed using an FE-SEM device (SUPRA-40VP, Carl Zeiss, Center for University-wide Research Facilities at Jeonbuk National University). XRD patterns were obtained using a high-resolution X-ray diffractometer (D8 Advance, Bruker). The XPS analysis, for exploring the surface composition of IZO thin films, was performed using a Theta Probe instrument (Thermo Fisher Scientific, Inc., USA). The STEM/EDS elemental mapping was carried out by using Cs₂-corrected-field emission transmission electron microscope/STEM (JEM-ARM200F, JEOL, Japan).

Electrical Characterization: The electrical performance of IZO-NF-based FETs was evaluated in air using a Keithley 4200A SCS (Tektronix, USA) connected to a probe station installed in a dark shielded box (MS TECH, South Korea) and a Keithley 2612B source meter (Tektronix, USA). For dielectric characterization, a 100-nm-thick Ag electrode was thermally evaporated onto the solution-processed Al₂O₃ layer using a shadow mask to form the metal–insulator–metal structure. The areal capacitance was measured using an LCR meter (ZM2376, NF Corporation). μ_{sat} and SS were calculated using Equations (6) and (7), respectively:

$$\mu_{\text{sat}} = \left(\frac{\partial \sqrt{I_{\text{DS}}}}{\partial V_G} \right)^2 \frac{2L}{WC_i} \quad (6)$$

where L, W, and C_i denote effective channel length, width, and dielectric areal capacitance, respectively.

$$SS = \frac{\partial V_G}{\partial \log_{10} I_D} \quad (7)$$

Ethylene gas sensing measurements were performed in a custom-designed chamber equipped with a heating stage. Prior to the measurement, the heating stage was heated to 200 °C, and nitrogen (N₂) gas was injected into the chamber using a mass flow controller to purge the chamber. N₂ facilitates the stabilization of the sensor system at elevated temperatures by purging ambient air from the chamber, thereby suppressing the reversible interaction between IZO oxide and oxygen molecules during ethylene exposure. Ethylene gas with various concentrations was injected into the chamber through a gas-tight syringe via a rubber septum. The change in resistance was observed using the LCR meter. The sensing response was determined using Equation (8):

$$\text{Response (\%)} = \frac{R_a - R_g}{R_a} * 100\% \quad (8)$$

where R_a is the initial resistance of sensor device, and R_g is the resistance recorded after the introduction of ethylene gas.

Supporting Information

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

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

amorphous oxide, chemiresistive gas sensors, electrospinning, field-effect transistors, semiconductor nanofiber

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