Performance improvement in electrospun InGaZnO nanofibres field-effect-transistors using low thermal budget microwave calcination and Ar/O₂ mixed-plasma surface treatment

Seong-Kun Cho & Won-Ju Cho*

In this study, we present a low thermal budget microwave annealing (MWA) method for calcination of electrospun In-Ga-ZnO (IGZO) nanofibres and demonstrate an improvement in the performance of IGZO nanofibre field-effect transistors (FETs) by Ar/O₂ mixed-plasma surface treatment. The IGZO nanofibres were fabricated by electrospinning method and calcined using MWA method. This process allowed for a significant reduction in the heat treatment temperature and time. Subsequently, plasma surface treatment using various ratios of Ar/O₂ gas mixtures was carried out. The surface morphology and chemical composition of MWA-calcined and plasma-treated IGZO nanofibres were studied by SEM and XPS analysis. In order to investigate the effects of MWA calcination combined with Ar/O₂ mixed-plasma treatment on the electrical properties and the reliability of nanofibres-based transistors, IGZO nanofibres FETs were fabricated and applied to resistor-loaded inverters. Our results show that the O₂ plasma treatment significantly improves the performance of IGZO nanofibres FETs and the resistor-loaded inverters based on IGZO nanofibres FETs, whereas Ar plasma treatment degrades the performance of these devices. The instability tests using positive bias temperature stress (PBTS) and negative bias temperature stress (NBTS) revealed that the O₂ plasma treatment contributed to the stability of IGZO nanofibres FETs. Our results suggest that the MWA calcination combined with the Ar/O₂ mixed-plasma surface treatment is a promising technique for the fabrication of high performance IGZO nanofibres FETs with low thermal budget processes.

Recently, the application of amorphous oxide semiconductors (AOS) as backplanes for the thin film transistors (TFTs) of active matrix liquid crystal displays (AMLCDs) and active matrix organic light emitting diode displays (AMOLEDs) has been an actively researched topic owing to its advantages over the currently existing technology. Amorphous indium gallium zinc oxide (a-IGZO) TFTs exhibit higher mobility than amorphous silicon (a-Si:H) TFTs, and they possess a superior uniformity compared with the polycrystalline silicon (poly-Si) TFTs because of their amorphous structure. In addition, a-IGZO has a wide band gap and high transmittance in the visible region, which makes it easy to access transparent optoelectronics. Despite these advantages, achieving a desired flexibility and stretchability in a-IGZO films still remains challenging. In addition, IGZO deposited by vacuum equipment, such as radio frequency (RF) magnetron sputtering, chemical vapor deposition (CVD) or atomic-layer-deposited (ALD), requires expensive equipment, and is disadvantageous for long process time and large area deposition. Recent intensive efforts have been focussed on the fabrication of IGZO nanofibres for their applications in the next-generation electronics, which are more flexible and stretchable. Electrospinning is one of the most commonly used manufacturing methods of nanofibres owing to its low manufacturing cost and simple procedure that can be carried out in absence of vacuum, since it is solution process
O₂ mixed-plasma treated IGZO nanofibres show a further reduction in their diameter with an increase in the Ar atmosphere when IGZO/PVP composite nanofibres are exposed to the microwave radiation. In addition, Ar/O₂ (~ 770 nm) in the diameter of nanofibres was observed after microwave calcination, as shown in Fig. 2b. The reduction in diameter is caused by the etching of the IGZO nanofibres by the plasma treatment condition. The reduction in diameter occurs due to the release of low molecular weight minerals, such as pyrrolidone, into the atmosphere when IGZO/PVP composite nanofibres are exposed to the microwave radiation. In addition, Ar/O₂ mixed-plasma treated IGZO nanofibres show a further reduction in their diameter with an increase in the Ar ratio, as shown in Fig. 2c–g. The average diameter of electrospun IGZO nanofibres is shown in Fig. 2h. The diameter of the IGZO nanofibres shown in Fig. 2h was averaged from 30 nanofibres selected from SEM images for each plasma treatment condition. The reduction in diameter is caused by the etching of the IGZO nanofibres by the additional Ar plasma, as Ar ions increase in the gas ratio of the Ar/O₂ mixed-plasma treatment.

Results

Morphological properties of electrospun IGZO nanofibres. The experimental setup of the electrospinning apparatus is shown in Fig. 1a, which utilizes NE-1000 of New Era Pump System as the syringe pump. The syringe pump pressure was set as 0.2 ml/br for electrospinning the IGZO nanofibres. A voltage of 20kV was applied to the needle to initiate the jet, and a grounded square copper plate (15 × 15 cm²) was used as a collector for nanofibres. The temperature and humidity were maintained at 25 °C and 25%, respectively. The entire process took approximately 2–4 minutes to ensure an adequate amount of sample collection. Figure 1b shows a schematic diagram of the fabrication process of IGZO nanofibres FETs. IGZO nanofibres were electrospun on a p-type Si (100) substrate with a 100-nm-thick thermally grown SiO₂ gate insulator layer. Then, the calcination of IGZO nanofibres was performed in ambient air for 2 min using a 1000 W microwave irradiation system with a frequency of 2.45 GHz. Subsequently, IGZO nanofibres were exposed to the plasmas of Ar/O₂ gas mixture with different flow rates in sccm (50/0, 40/10, 25/25, 10/40, 0/50) for removing carbon-based impurities and improving their electrical properties.

Figure 2 shows SEM images of as-electrospun and plasma treated IGZO nanofibres with the average diameter of the nanofibres after each process. The as-spun IGZO/polyvinylpyrrolidone (PVP) composite nanofibres display a uniform morphology (Fig. 2a) with an average diameter of approximately 960 nm. A remarkable decrease (~770 nm) in the diameter of nanofibres was observed after MWA calcination, as shown in Fig. 2b. The reduction in the diameter occurs due to the release of low molecular weight minerals, such as pyrrolidone, into the atmosphere when IGZO/PVP composite nanofibres are exposed to the microwave radiation. In addition, Ar/O₂ mixed-plasma treated IGZO nanofibres show a further reduction in their diameter with an increase in the Ar ratio, as shown in Fig. 2c–g. The average diameter of electrospun IGZO nanofibres is shown in Fig. 2h. The diameter of the IGZO nanofibres shown in Fig. 2h was averaged from 30 nanofibres selected from SEM images for each plasma treatment condition. The reduction in diameter is caused by the etching of the IGZO nanofibres by the additional Ar plasma, as Ar ions increase in the gas ratio of the Ar/O₂ mixed-plasma treatment.

Electrical properties of IGZO nanofibres FETs. Since Ar and O₂ plasma treatments may result in enhancement or degradation of the performance of IGZO nanofibres FETs depending on the treatment conditions, it is essential to establish the optimized plasma treatment conditions for maximizing the device performance. Figure 3 illustrates (a) transfer curves (Iₘ–Vₛ) and (b) output curves (Iₘ–Vₚ) of IGZO nanofibres FETs treated with various ratios of Ar/O₂ mixed-plasma. The device performance was found to be strongly dependent on the gas ratios of Ar/O₂ mixed plasma. As the ratio of O₂ in the Ar/O₂ mixed-gas flow increases, the drain current increases. The maximum drain current is observed at the Ar/O₂ flow rate (sccm) ratio of 0/50 (pure oxygen...
atmosphere). In contrast, the drain current of the nanofibres FET decreases with an increase in the Ar ratio of the Ar/O2 mixed-gas flow. For the Ar/O2 flow rate (sccm) of 40/10 and 50/0 (higher Ar content), the electrical properties of the device deteriorated compared with the ones without plasma treatment. The corresponding electrical parameters extracted from ID-VG curves (Fig. 3) are summarized in Table 1. As the O2 flow rate increases in the Ar/O2 mixed-gas, an improvement in the on/off current ratio (Ion/Ioff), the threshold voltage (Vth), the field effect mobility (μFE), the sub-threshold swing (S.S.), and the interface trap density (Dit) is observed. Conversely, a deterioration of the electrical parameters is observed with an increase in the Ar flow rate. Therefore, our results indicate that the higher O2 gas content used in the Ar/O2 mixed-gas plasma treatment yields better electrical characteristics of the IGZO nanofibres FETs.

After fabricating high performance electrospun IGZO nanofibres FETs through a combination of low thermal budget MW A calcination and Ar/O2 mixed-plasma surface treatment processes, we integrated the IGZO nanofibres FETs into resistor-loaded inverters. The typical voltage transfer curves (VOUT-VIN) of the resistor-loaded inverter based on IGZO nanofibres FETs are shown in Fig. 4a. Evidently, the output voltage (VOUT) of all inverters is inverted from the input voltage (VIN). The VOUT level for the low VIN was approximately equal to the supply voltage VDD = 10 V. However, it should be noted that as the ratio of O2 in the Ar/O2 mixed-gas flow increases, the VOUT level for the high VIN approaches 0 V. Figure 4b shows the corresponding gain (defined as -dVout/dVin) extracted from the VOUT-VIN curves with the schematic of the circuit. The resistor-loaded inverter plasma-treated at Ar/O2 flow rate (sccm) ratio 0/50 (pure oxygen atmosphere) shows a maximum gain of 3.0; however, the gain decreases with increasing Ar ratio in the mixed-gas flow. Figure 4c shows the dynamic responses of the resistor-loaded inverter at VDD = 10 V when VIN is pulsed at 1 Hz. The inverter shows the closest behaviour (operation and response) to the square wave input signal under the mixed plasma flow rate (sccm) of Ar/O2 = 0/50. Therefore, we believe that the MW A calcination and Ar/O2 mixed-plasma treatment with low thermal budgets enhance the operational stability of IGZO nanofibres FETs.

Reliability evaluation of IGZO nanofibres FETs. Although nanofibres structures have the advantages of being transparent, flexible, and stretchable, the extended use of nanofibres-based electronic devices can lead to instability of IGZO nanofibres FETs. In particular, the reliability of oxide semiconductor-based transistors is the most desired trait for practical applications26,27. To this end, several research groups have studied the reliability of IGZO TFTs; however, there has been no report on the reliability of IGZO nanofibres FETs in the literature. Therefore, we evaluated the electrical stability of the IGZO nanofibres FETs by observing the change in the threshold voltage (∆Vth) under prolonged gate bias stress and studied the effect of plasma treatment on the mixing ratio of Ar/O2. Figure 5 shows the ∆Vth of IGZO nanofibres FETs measured for 103 seconds using PBTS (VG = Vth0 + 20 V) and NBTS (VG = Vth0 − 20 V) tests at 25, 55, and 85 °C. It is evident (from Fig. 5) that the threshold voltage Vth0 of the non-plasma treated device shifts in the positive or negative direction depending on the polarity of the gate stress voltage. This is caused by the trapping of electrons or holes in the trap states, where ∆Vth measured during the PBTS and NBTS tests are affected by the oxygen related trap states (acceptor-like) and the oxygen vacancy related trap states (donor-like), respectively26–28. The Ar/O2 mixed-plasma treatment reduces the ∆Vth of the IGZO nanofibres FETs. In particular, a smaller value of ∆Vth was observed with an increase in the O2 gas ratio in the mixed-plasma. For each device, ∆Vth increases with the stress time and temperature, and
the rate of increase is dependent on the stress temperature, as shown in Fig. 5a–c. A summary of measured ΔVth after 10^3 seconds of PBTS and NBTS tests for various Ar/O2 mixed-plasma treated IGZO nanofibres FETs at 25, 55, and 85 °C is shown in Table 2.

The ΔVth data in Fig. 5a–c was fitted using the following stretched-exponential equation for the charge trapping states:\[\text{ΔVth} = \Delta V_{\text{th0}} \times e^{-\left(\frac{t}{\tau}\right)^{\beta}}\]

Table 1. Electrical parameters of IGZO nanofibres FETs for various Ar/O2 mixed-plasma flow rates.

| Ar/O2 mixed-gas flow [sccm] | Ion/Ioff | Vth [V] | μFE [cm²/V·s] | S.S [mV/dec] | Dit [cm⁻²] |
|-----------------------------|----------|---------|---------------|-------------|-----------|
| 50/0                        | 0.06 × 10⁶ | 5.3    | 0.43          | 735.2       | 2.67 × 10¹² |
| 40/10                       | 0.36 × 10⁶ | 2.7    | 1.92          | 405.8       | 1.47 × 10¹² |
| 25/25                       | 1.10 × 10⁶ | 2.1    | 3.68          | 351.7       | 1.27 × 10¹² |
| 10/40                       | 2.42 × 10⁶ | 1.8    | 4.23          | 315.4       | 1.14 × 10¹² |
| 0/50                        | 4.35 × 10⁶ | 0.9    | 5.32          | 282.1       | 1.02 × 10¹² |
| w/o plasma                  | 0.46 × 10⁶ | 5.6    | 0.82          | 346.6       | 1.26 × 10¹² |

Figure 2. SEM images of IGZO nanofibres (a) as-spun at 5,000 times magnifications, (b) without plasma treatment, and with Ar/O2 mixed-plasma treatment under gas flow rates (in sccm) in the ratio of (c) 50/0, (d) 40/10, (e) 25/25, (f) 10/40, and (g) 0/50. (h) Average diameter of the electrospun IGZO nanofibres with plasma treatment at 30,000 times magnifications.

Figure 3. (a) Transfer curves and (b) Output curves of IGZO nanofibres FETs treated with various flow rate (in sccm) ratios of Ar/O2 mixed-plasma.
\[ \Delta V_{th} = \Delta V_{th0} \exp \left[ -\left( \frac{t}{\tau} \right)^{\beta} \right] \]  

\[ \tau = \tau_0 \exp \left( \frac{E_{\tau}}{k_B T} \right) = \nu^{-1} \exp \left( \frac{E_{\tau}}{k_B T} \right) \]  

where \( \Delta V_{th0} \) is the threshold voltage shift at an infinite time, \( \tau \) is the characteristic trapping time of the charge carrier, and \( \beta \) is the stretched-exponential exponent. As shown in Fig. 5, the fitting curves are in good agreement with the experimental results, suggesting that \( \tau \) depends on temperature, and the change in \( V_{th} \) occurs through the thermal activation process. In the stretched-exponential equation, the thermal activation of \( \tau \) of electrons follows:  

\begin{table}[h]
\centering
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline
\textbf{Ar/O}_2 [sccm] & \multicolumn{6}{|c|}{\textbf{w/o plasma}} \\
\hline
\textbf{PBTS @ 1000 s} & \textbf{25°C} & 8.0 & 7.5 & 7.0 & 6.4 & 5.3 & 4.7 \\
\hline
& \textbf{55°C} & 12.6 & 10.2 & 9.5 & 8.9 & 8.2 & 7.6 \\
\hline
& \textbf{85°C} & 15.6 & 14.1 & 13.5 & 13.2 & 12.5 & 9.5 \\
\hline
\textbf{NBTS @ 1000 s} & \textbf{25°C} & -3.5 & -2.3 & -1.8 & -1.5 & -1.1 & -0.7 \\
\hline
& \textbf{55°C} & -6.6 & -6.3 & -4.5 & -4.3 & -4.1 & -2.6 \\
\hline
& \textbf{85°C} & -9.4 & -9.3 & -7.2 & -6.3 & -5.1 & -3.6 \\
\hline
\end{tabular}
\caption{\( \Delta V_{th} \) after 10^4 seconds of PBTS and NBTS tests for various Ar/O\_2 mixed-plasma treated IGZO nanofibres FETs measured at 25, 55, and 85 °C.}
\end{table}
In Fig. 6, the Arrhenius plots display the relationship between the logarithm of the characteristic trapping time $\ln(\tau)$ and the inverse of the temperature $1/T$. Figure 6a and b correspond to the PBTS and NBTS tests, respectively, for various Ar/O$_2$ mixed-plasma treated IGZO nanofibres FETs. As evident from Fig. 6, the linear relationship between $\ln(\tau)$ and $1/T$ indicates a thermally activated charge trapping process in these devices. The slope of the Arrhenius plot represents $E_\tau$ for the charge transport under the PBTS and NBTS tests. Table 3 summarizes the estimated values of $E_\tau$ under the PBTS and NBTS tests performed on the IGZO nanofibres FETs treated with various ratios of Ar/O$_2$ mixed-plasma. Compared with the non-plasma treated device, the Ar/O$_2$ mixed-plasma treated devices exhibited lower $E_\tau$. Particularly, a lower value of $E_\tau$ was observed for the devices treated with higher O$_2$ gas ratio in the Ar/O$_2$ mixed-gas flow. The smallest values of $E_\tau$ (~0.29 eV for PBTS and ~0.50 eV for NBTS) and the highest stability was obtained for the IGZO nanofibres channels treated with the Ar/O$_2$ flow rate (sccm) ratio of 0/50 compared with other channels. These results are consistent with those displayed in Figs. 3 and 4.

Figure 7 shows the XPS O1s spectra to evaluate the chemical states of IGZO nanofibres after the Ar/O$_2$ mixed-plasma treatment with the flow rate (sccm) ratios of 50/0 (Fig. 7a) and 0/50 (Fig. 7b). The XPS spectra were collected to a depth of few-nanometres beyond the etched surface of IGZO nanofibres by Ar ions for avoiding any surface contamination. Unlike thin films of uniform thicknesses, the IGZO nanofibres have several gaps between the fibres as shown in Fig. 2; therefore, the components of the underlying SiO$_2$ gate insulator are also observed in the XPS spectra. Thus, we have deconvoluted the O1s spectra into four individual component peaks. The peaks observed at 529, 530, and 531 eV are related to the components of the IGZO nanofibres, whereas the peaks at 532 eV is associated with the underlying SiO$_2$ gate insulator. The main peak at 529 eV represents the stoichiometric oxygen (M-O), the subpeak at 530 eV represents the oxygen vacancies (M-Ovac), and the subpeak at 531 eV is associated with the loosely bound oxygen impurities (M-OH) such as chemisorbed oxygen, H$_2$O, and CO$_3$. Since the Si-O peaks observed from the underlying SiO$_2$ gate insulators show a similar contribution in both spectra, we focus our discussion only on the M-O, M-Ovac, and M-OH bonds detected from the IGZO nanofibres. As evident from Fig. 7, the ratio of M-Ovac and M-OH bonds is smaller in the IGZO nanofibres treated with the Ar/O$_2$ mixed-plasma at a flow rate (sccm) ratio of 0/50 (purely oxygen atmosphere) compared with the ones treated at 50/0 flow rate. The oxygen vacancies of M-Ovac bonds are known to cause stability deterioration in gate bias temperature stress test$^{30}$, whereas the loosely bound oxygen impurities of M-OH bonds act as the charge trap states and reduce the on-current in IGZO films$^{31}$. Hence, we conclude that the mixed-plasma treated IGZO nanofibres FETs treated with Ar/O$_2$ flow rate (sccm) ratio of 0/50 exhibit better electrical properties and reliability due to the low impurity concentration compared with those treated at 50/0 flow rate.

In this study, we employed the low thermal budget MWA process for calcination of electrospun IGZO nanofibres and investigated the effects of Ar/O$_2$ mixed-plasma surface treatment on the performance of IGZO nanofibres-based FETs. The MWA process (at 2.45 GHz and 1000 W) allowed rapid calcination (in 2 min) of the electrospun IGZO nanofibres in the ambient air. Additionally, the plasma surface treatment with Ar/O$_2$ gas mixtures altered the surface morphology and chemical composition of the IGZO nanofibres, eventually affecting the electrical properties and reliability of the IGZO nanofibres FETs. Our results indicate that oxygen in the Ar/O$_2$
mixed-plasma contributes to the removal of impurities, whereas argon plays a role in reducing the diameter of the IGZO nanofibres. In addition, the higher oxygen content in the Ar/O2 mixed-plasma significantly improved the performance of the IGZO nanofibres FETs and resistor-loaded inverters based on IGZO nanofibres FETs. Conversely, the higher argon content was found to degrade the performance of these devices. Instability tests using PBTS and NBTS revealed that O2 plasma treatment contributed to the stability improvement of the IGZO nanofibres FETs. Therefore, we expect that the MW A calcination process combined with the Ar/O2 mixed plasma surface treatment is a promising low thermal budget approach for the fabrication of high performance IGZO nanofibres FETs.

Methods

Solution synthesis procedure. Solution of IGZO nanofibres was synthesized by the sol-gel reaction of precursors. Indium nitrate hydrate [In(NO3)3·xH2O], gallium nitrate hydrate [Ga(NO3)3·xH2O], and zinc acetate dehydrate [Zn(CH3COO)2·2H2O] were used as precursors for the sol-gel reaction. These precursors were dissolved in N, N-dimethylformamide (DMF) solvent in a molar ratio of 2:1:1, and the solution was mixed at room temperature (25 °C) for 2 hours using a stirrer. Then, 2.5 ml of ethanol containing 0.18 g of polyvinylpyrrolidone (PVP, MW ≈ 1300000) was added to the solution, and it was mixed at room temperature (25 °C) for 2 hours using a stirrer.

Device fabrication procedure. A 100-nm SiO2 gate insulator is thermally grown and RCA cleaned on the p-type Si (100) substrate. Subsequently, a channel layer was formed by spinning IGZO nanofibres using the electrospinning method. To remove the polymer matrix, the MW A calcination annealing was performed by using a 1000 W microwave irradiation system with a frequency of 2.45 GHz in the ambient air for 2 min. Then, the active channel region of the IGZO nanofibres FETs was defined by photolithography and wet-etching technique using a 30:1 ratio of buffer oxide etchant (BOE). The width of the defined active channel region is 10 μm, and the length is 20 μm. Further, the IGZO nanofibres were exposed to the plasma of various Ar/O2 mixed-gas flow rates (in sccm; 50/0, 40/10, 25/25, 10/40, 0/50) in a reactive-ion etching (RIE) system. The power of the plasma treatment, the forward pressure, and the exposure time were selected as 200 W, 300 mTorr, and 20 s, respectively. To improve the electrical properties, the PDA process was performed, involving the conventional thermal annealing (CTA) in a furnace at 600 °C in O2-atmosphere for 30 min. Finally, IGZO nanofibres FETs were fabricated by depositing a 150 nm-thick Ti layer using e-beam evaporator and forming the source and drain electrodes of FETs through the lift-off process.

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Author contributions

S.K. Cho wrote the main manuscript text and prepare the figure and W.J. Cho reviewed the manuscript. Both S.K. Cho and W.J. Cho have materially participated in the research and approved the final article.

Competing interests

The authors declare no competing interests.

Additional information

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