

Article

Improved Negative Bias Stress Stability of Sol–Gel-Processed Li-Doped SnO₂ Thin-Film Transistors

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Abstract: In this study, sol–gel-processed Li-doped SnO₂-based thin-film transistors (TFTs) were fabricated on SiO₂/p+ Si substrates. The influence of Li dopant (wt%) on the structural, chemical, optical, and electrical characteristics was investigated. By adding 0.5 wt% Li dopant, the oxygen vacancy formation process was successfully suppressed. Its smaller ionic size and strong bonding strength made it possible for Li to work as an oxygen vacancy suppressor. The fabricated TFTs consisting of 0.5 wt% Li-doped SnO₂ semiconductor films delivered the field-effect mobility in a 2.0 cm²/Vs saturation regime and I_{on}/I_{off} value of 1 × 10⁸ and showed enhancement mode operation. The decreased oxygen vacancy inside SnO₂ TFTs with 0.5 wt% Li dopant improved the negative bias stability of TFTs.



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Keywords: sol–gel; Li doping; thin-film transistor; SnO₂; negative bias stability

1. Introduction

Recently, the demand for oxide semiconductors has been increased due to their high usability. Oxide semiconductor-based thin-film transistors (TFTs) show higher mobility than amorphous phase Si and can achieve higher stability than low-temperature polysilicon material-based transistors [1–5]. In addition, their wide bandgap properties are critical factors to realize transparent electronics. These advantages are being used not only for large-area displays but also for several devices that require transparent features. The most commercially available oxide semiconductors are the indium gallium zinc oxide (IGZO) semiconductors for channel materials of TFTs. However, IGZO contains indium, which has low price competitiveness because it is a rare-earth element [6]. Therefore, many studies have been conducted to fabricate inexpensive oxide TFTs to replace IGZO, which is expensive, and ZnO, ZnSnO, and SnO₂, excluding indium, have shown excellent performance as substitutes [7–9].

SnO₂ is a promising material for use as an active channel layer of high-performance oxide TFT (high intrinsic Hall mobility, transparency because of a wide bandgap, and a low melting point, which determines the sintering temperature for achieving high-quality crystalline thin films [10–12]. To date, the expensive and time costly vacuum-based deposition technique has been used for fabricating metal–oxide-based thin films. To overcome this issue, the sol–gel process has been recently employed [13–16]. The sol–gel route process is cost-effective, eco-friendly, and adaptable for large area applications. Moreover, because the starting materials used in this process are in the liquid phase, they have been considered promising inks for various printing systems [17]. Although the abovementioned sol–gel process-based electronics have shown high performance, one critical disadvantage is their chemical or bias stress instability, originating from the absorbed molecules or oxygen vacancy inside metal–oxide semiconductors (MOs), which

leads to threshold voltage instability. To solve this issue, an oxygen vacancy suppressor has been used [18,19]. In this study, by producing $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ and ethanol-based precursors with a Li dopant, Li-doped SnO_2 TFTs are realized by the sol–gel process. The optimized Li dopant works as a promising oxygen vacancy suppressor to reduce oxygen vacancy formation inside SnO_2 active channel layers. First, the structural, chemical component, and optical characteristics of Li-doped SnO_2 thin films are investigated. The electrical characteristics of Li-doped SnO_2 -based TFTs are also investigated.

2. Materials and Methods

Tin (II) chloride dehydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, Sigma Aldrich, St. Louis, MO, USA) was used as a precursor. Tin (II) chloride dehydrate was dissolved into ethanol in an air ambient and at room temperature. The three precursor solutions were made by mixing SnCl_2 with ethanol at 0.030 M (0.0677 g per 10-mL ethanol). One solution was made for reference, and the other two were mixed with 0.5- and 1.0-wt% Li. Lithium acetate dehydrate ($\text{C}_2\text{H}_3\text{LiO}_2 \cdot 2\text{H}_2\text{O}$, Sigma Aldrich) was used as a source of a Li dopant. A coplanar structure (bottom gate and bottom source/drain) was fabricated with Si/ SiO_2 substrates. Highly doped p-type Si was used as a gate electrode. Thermally grown 100 nm-thick SiO_2 was used as an insulator, and Au was used for the source/drain electrode. The Au source/drain electrodes were formed by e-beam evaporation and using the lift-off fabrication process. The length and width of the channel were 100 and 1000 μm , respectively. The substrates were cleaned for 1 h in ultraviolet (UV)/ O_3 (SENlights SSP16-110) to eliminate organic impurities and improve adhesion properties. The prepared precursors were coated onto the UV-cleaned substrates at 3000 rpm for 50 s. To evaporate ethanol, the coated substrates were baked at 150 °C on a hot plate (CORNING PC-420D) for 10 min. After prebaking, the substrates were immediately placed in a furnace tube (U1Tech PTF-1203) and the temperature was gradually increased 500 °C. Thereafter, the substrates were annealed for 2 h. The phase and structural properties of the TFTs were analyzed by measuring Grazing Incidence X-ray Diffraction (GIXRD, A Philips X'pert Pro, Philips, Amsterdam, The Netherlands). Ultraviolet–visible spectroscopy (UV–vis, LAMBDA 265) was used to obtain the optical properties of the fabricated TFTs. The film thickness of the film was measured using a scanning probe microscope (Park NX20, tapping mode). The chemical properties of the TFTs were analyzed using X-ray photoelectron spectroscopy (XPS: ULVAC-PHI), and their electrical properties were measured using a Keithley 2636B semiconductor parameter analyzer (Keithley Instruments, Cleveland, OH, USA) and a probe station (MST T-4000A).

3. Results and Discussion

Figure 1 shows the GIXRD spectra of the sol–gel-processed SnO_2 films with various Li contents. All the deposited TFTs showed the polycrystalline tetragonal SnO_2 phase. The diffraction peaks at 26.6, 33.8, 37.95, and 51.8 corresponded to the crystal planes of (110), (101), (200), and (211) (JCPDS card number: 41-1445), respectively. The peak at 31.7 corresponded to Sn (JCPDS card number: 82-2958), indicating that Sn was partially oxidized. The full width at half-maximum (FWHM) of each diffraction peak was associated with the crystalline size of the films. The (110) plane was primarily the direction of growth of SnO_2 films. The crystalline size of the films under the (110) crystal plane was calculated using the Scherrer equation:

$$D = \frac{0.9\lambda}{\beta \cos \theta}, \quad (1)$$

where D , λ , β , and θ indicate the crystalline size, $\text{CuK}\alpha$ wavelength (1.54 Å), FWHM of the peak, and peak position, respectively. The obtained crystalline sizes of the SnO_2 TFTs from the (110) plane were 1.33 nm, 1.47 nm, and 1.47 nm for Li-doped SnO_2 films with Li contents of 0.0 wt%, 0.5 wt%, and 1.0 wt%, respectively. The Li-doped SnO_2 films exhibited larger crystalline sizes than the undoped SnO_2 films. The added Li could accelerate the growth process during the sol–gel process [20].

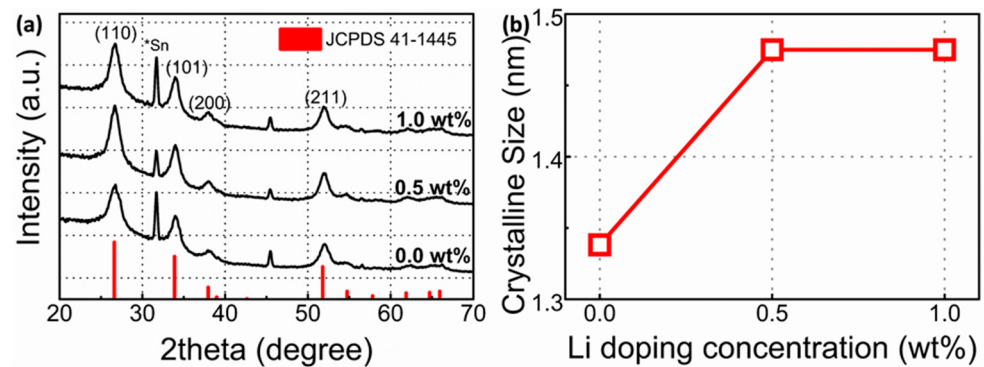


Figure 1. (a) The Grazing Incidence X-ray Diffraction spectra of SnO₂ films and (b) calculated crystalline sizes of SnO₂ films as a function of Li (wt%).

Figure 2a–c show the representative O 1s XPS spectra of Li-doped SnO₂ as a function of Li (wt%). The O 1s peak was divided into three types of peaks [21]. One peak is for the O₂[−] combined with the adjacent ion in Lattice (O_{Lattice}), another for the Oxygen bonds related to the oxygen vacancy (O_{Vacancy}), and the other for the amount of OH. Figure 2d shows that the variation in the composition ratio of O_{Vacancy}, O_{Lattice}, and −OH groups as a function of Li (wt%). When a 0.5 wt% Li dopant was added, the relative percentage of O_{Vacancy} gradually decreased from 27.5% to 21.7%, whereas the relative percentage of O_{Lattice} gradually increased from 65.7% to 68.1%, which meant that a small amount of Li dopant could work as a carrier inhibitor. Several factors, such as electronegativity, standard electrode potential (SEP), and ion radius, need to be assessed for dopants to be used as carrier inhibitors [22,23]. The electronegativity of dopants and oxygen should significantly differ from each other. When the SEP of the dopant is lower than that of the host, the dopant–oxygen bond becomes stronger. The electronegativity and SEP of Sn hosts and Li dopants were 1.96 and −0.14 V and 0.98 and −3.04 V, respectively. Based on the aforementioned theory, the Li dopant could work as a carrier inhibitor successfully. Moreover, the radius of the dopant and host ions should not differ considerably to avoid the breakdown of the structure; the radii of Sn⁴⁺ and Li⁺ were 74 and 68 pm, respectively, making Li⁺ suitable as the dopant. However, when 1.0-wt% Li was added, the relative percentage of O_{Vacancy} increased from 21.2% to 23.3%, while the relative percentage of O_{Lattice} decreased from 68.1% to 66.6%.

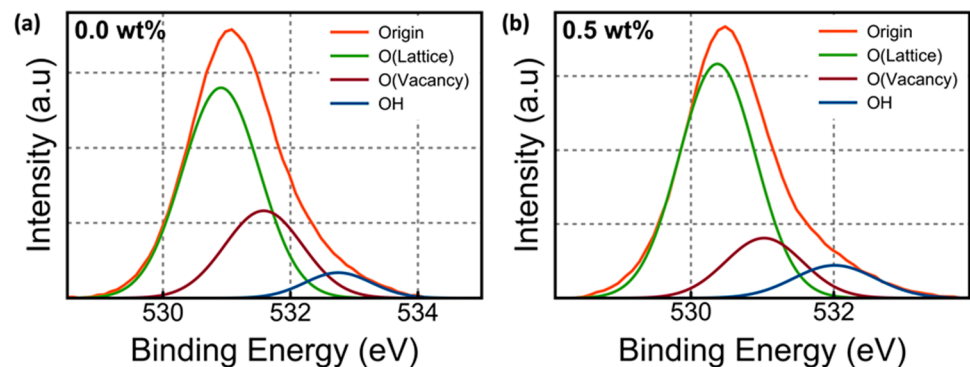


Figure 2. Cont.

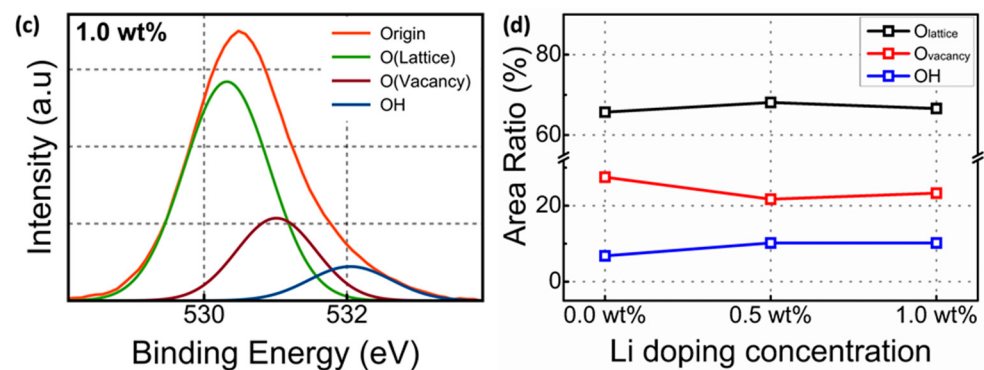


Figure 2. (a–c) O 1s XPS spectra and O 1s composition area ratio of Li-doped SnO₂ and (d) the variation of the composition ratio of O_{Vacancy}, O_{Lattice}, and –OH groups as a function of Li (wt%).

To elucidate the effect of the Li dopant amount on the optical bandgap of SnO₂ films, the transmittance plots of the SnO₂ films as a function of Li dopant amounts were obtained using a UV–vis spectrophotometer. Regardless of the Li dopant amount all the fabricated SnO₂ films showed a transmittance of >97.5% in the visible range (380–900 nm) (Figure 3a). However, at less than 350 nm, all the SnO₂ films showed a drastic decrease in the bandgap absorption, corresponding to the optical bandgap of SnO₂ films. The optical bandgap can be obtained by extrapolating the line segment in Figure 3b and using the following equation:

$$(\alpha h\nu)^{1/n} = A(h\nu - E_g), \quad (2)$$

where α is the absorption coefficient, A is a constant, and E_g is the optical bandgap of SnO₂ films. For direct bandgap transition, the n value is $\frac{1}{2}$. Using Equation (2), the estimated optical bandgaps of each film were 3.79 eV, 3.87 eV, and 3.93 eV. As a function of Li concentration, the optical bandgap of SnO₂ films increased. Regardless of the Li dopant amount, the obtained optical bandgap of the fabricated SnO₂ films was larger than that of bulk SnO₂ (3.60 eV). If the film thickness was close to the Bohr radius owing to the quantum confinement effect, the optical bandgap could increase. The change in the optical bandgap can be calculated using the following equation [24,25]:

$$\Delta E_g = \frac{h^2}{8t^2} \left(\frac{1}{m_e^*} + \frac{1}{m_h^*} \right), \quad (3)$$

where h is the Plank's constant, t is the thickness of SnO₂ films, m_e^* and m_h^* are the effective masses of electrons and holes, respectively (for SnO₂, $m_e^* = 0.275m_0$, $m_0 = 9.1 \times 10^{-31}$ kg). Because $m_e^* \ll m_h^*$, $1/m_h^*$ term can be ignored [24]. Regardless of Li dopant amount, the film thickness was approximately 5.5 nm, and the calculated optical bandgap was 3.90 eV. An additional change in the optical bandgap could be attributed to the free carrier concentration. Figure 3c presents the valence band (VB) spectra of Li-doped SnO₂ films using XPS. The VB offset values between the maximum VB value and the Fermi level were extrapolated by linear fitting and the values were 3.65 eV, 3.46 eV, and 3.65 eV, respectively [26]. Using the obtained bandgap and VB offset values, the conduction band (CB) offset values between the minimum of CB value and Fermi level could be calculated. The calculated CB offset values were 0.14 eV, 0.41 eV, and 0.28 eV, respectively, and they were proportional to the carrier concentration. The CB offset value of the 0.5 wt% Li dopant was the lowest, implying the lowest carrier concentration among the three cases by suppressing O_{Vacancy} formation, determines the free carrier concentration in the MOS system [27].

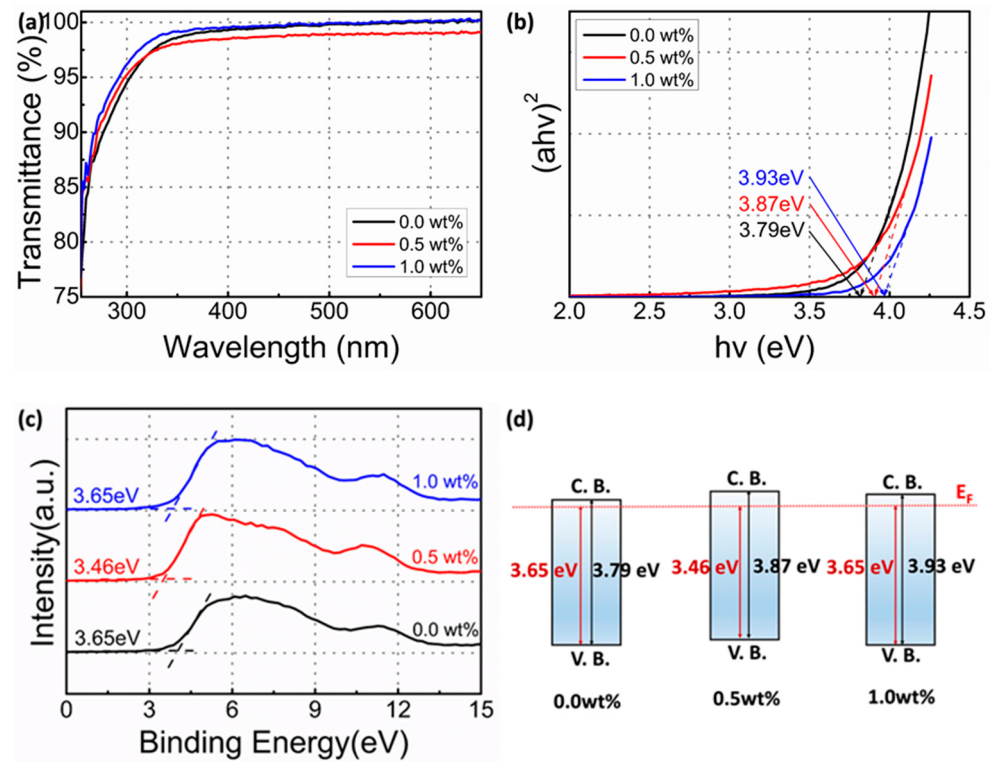


Figure 3. (a) Transmittance spectra of Li-doped SnO₂ films as a function of Li (wt%). (b) Optical bandgap of Li-doped SnO₂ films as a function of Li (wt%). (c) VB offset based on XPS spectra. (d) Schematic energy band based on the Fermi energy-level position.

Figure 4 shows the electrical characteristics of the fabricated Li-doped SnO₂ based TFTs represented by output and transfer curves. The output curve (Figure 4a–c) was probed every 10 V between the gate voltage -30.0 and 30.0 V. Regardless of the Li dopant amounts, all the fabricated SnO₂ TFTs showed the conventional n-type semiconductor properties and exhibited a high on/off current ratio (over 10^7). The relatively large work function of an Au source and a drain electrode on n-type semiconductor results in the formation of Schottky contacts. The nonlinear relationship between drain currents and voltages originates from the abovementioned energy barrier between electrodes and semiconductors [14]. The TFT fabricated using pure SnO₂ showed the depletion-mode operation with a negative threshold voltage (V_{th}). This device, which is in the depletion mode, could easily form a channel at a gate voltage of 0.0 V owing to the presence of excessive free carriers in the SnO₂ TFT. However, 0.5- and 1.0-wt% Li-doped TFTs showed the enhancement-mode operation with a positive V_{th} . Compared with pure SnO₂, the reduced $O_{vacancy}$ generated less free carriers, leading to a late turn on. Field effect mobility (FEM) in the saturation regime and V_{th} were obtained by linear fitting to the $(I_D)^{0.5}-V_G$ plot using the following equation:

$$I_D = \mu C_i \frac{W}{2L} (V_G - V_{th})^2 \quad (4)$$

where C_i (insulator capacitance) = 3.45×10^{-8} F/cm², W (channel width) = 1000 μ m, L (channel length) = 100 μ m. The FEM in the saturation regime of the SnO₂ TFT was 5.91 cm²/Vs. The FEM values of the TFTs consisting of 0.5- and 1.0 wt% Li doped SnO₂ TFTs were 2.0 and 4.42 cm²/Vs, respectively. Grain boundary scattering, related to the grain boundary number, inside the channel material was strongly affected the FEM of TFTs. For example, a transistor, consisting of channel material, having large crystallinity, showed increased FEM by suppressing the grain boundary scattering [28]. Although based on the GIXRD data, the calculated crystalline size of the pure SnO₂ films was smaller than those of the Li-doped SnO₂ films, the FEM in saturation regime of pure SnO₂ TFTs

showed the highest values. For the MOS system, the main transportation mechanism was the percolation conduction mechanism. Based on this mechanism, the FEM could be improved by filling trap sites at high carrier concentrations [29]. The number of free carriers could be increased by forming O_{Vacancy} inside MOSs. O_{Vacancy} worked as donor levels and generated carriers using the following equation:



The decreased FEM value of 0.5-wt% Li-doped SnO_2 -based TFT attributed to the decrease in the number of O_{Vacancy} , which was confirmed using XPS data. This is a critical factor, compared with grain boundary scattering effects in this system. Additionally, The reduced number of O_{Vacancy} was the reason for the low FEM value of the 0.5-wt% Li-doped SnO_2 -based TFTs compared with that of pure SnO_2 -based TFTs [16]. The 1.0 wt% Li-doped SnO_2 based TFTs again showed increased O_{Vacancy} again, and which increased the FEM and induced a negative shift of V_{th} compared with the 0.5 wt% Li-doped based SnO_2 TFTs. It was already reported that Li can be located at the interstitial site and substitutional site in the SnO_2 matrix. If Li is located at the interstitial site, this can increase carrier concentration by generating on electron. The increased carrier concentration results in the enhanced FEM, based on aforementioned the percolation conduction mechanism. However, if Li is located at a substitutional site in the SnO_2 matrix, it can neutralize the electrons, which are the system's main carriers; because of fewer electrons, FEM decreases.

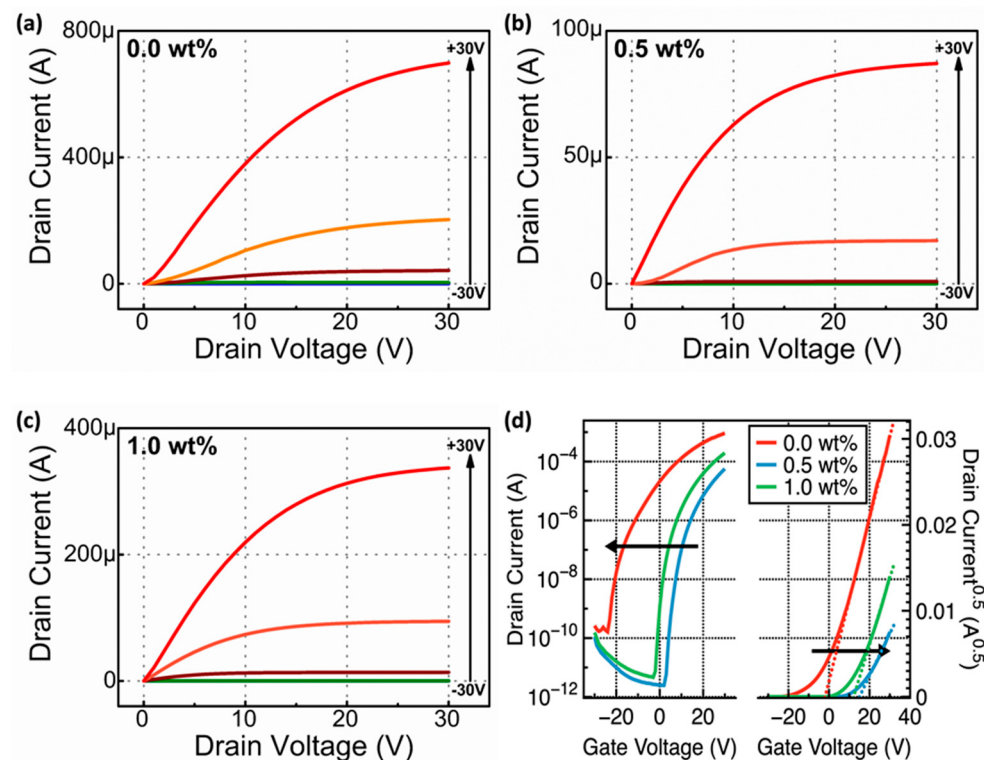


Figure 4. (a–c) I_D – V_D curves and (d) transfer curves and $I_D^{0.5}$ vs. V_G curves of Li-doped SnO_2 based TFTs, as Li (wt%) increases.

Figure 5a–c show the transfer curves of the representative TFT as a function of the negative bias stress (NBS) time with a drain voltage of +5.0 V. During the NBS test, -30.0 V was biased to gate electrodes for 1000 s. Under these NBS conditions, V_{th} shifts -10.5 , 1.32 , and 3.81 V , respectively. The extracted ΔV_{th} (5 devices) is plotted in Figure 5d to check the statistical distribution. The 0.5 wt% Li-doped based SnO_2 based TFT exhibited the lowest V_{th} shift among the three cases. Based on XPS data, 0.5 wt% Li-doped SnO_2

based TFT showed the lowest O_{Vacancy} concentration. Based on the percolation conduction mechanism, the lowest O_{Vacancy} leads to the lowest FEM and improved NBS properties. This negatively shifted V_{th} originated from the tapping of positive charges at the interface between semiconductors and insulators. O_{Vacancy} could form V_{O}^{2+} and $2e^-$. V_{O}^{2+} could move to the interface between semiconductors and insulators during the NBS test. By adding the optimal amount Li dopant, the O_{Vacancy} could be decreased by the formation of Li- O_{Vacancy} pairs, leading to improved NBS properties [18,19,30]. In addition, compared with sol-gel-processed Mg- and Ti-doped based SnO_2 , Li-doped SnO_2 based TFTs show more stable NBS properties [19,30]. Comparing to the previous results, Li-doped SnO_2 TFTs show similar or higher field effect mobility and comparable negative bias stability. The reported Si- and Ga-doped SnO_2 based TFTs show higher FEM and the Si-doped SnO_2 based TFTs show the better NBS properties [31]. Corresponding to deposition method, the crystallinity, chemical composition, and carrier concentration were changed. In this paper, the sol-gel-processed SnO_2 -based thin film transistors show lower field effect mobility, compared to reported SnO_2 semiconductors deposited by the vacuum-based deposition technique. However, sol-gel-processed Li doping can be applied to other types of the solution-processed oxide TFTs.

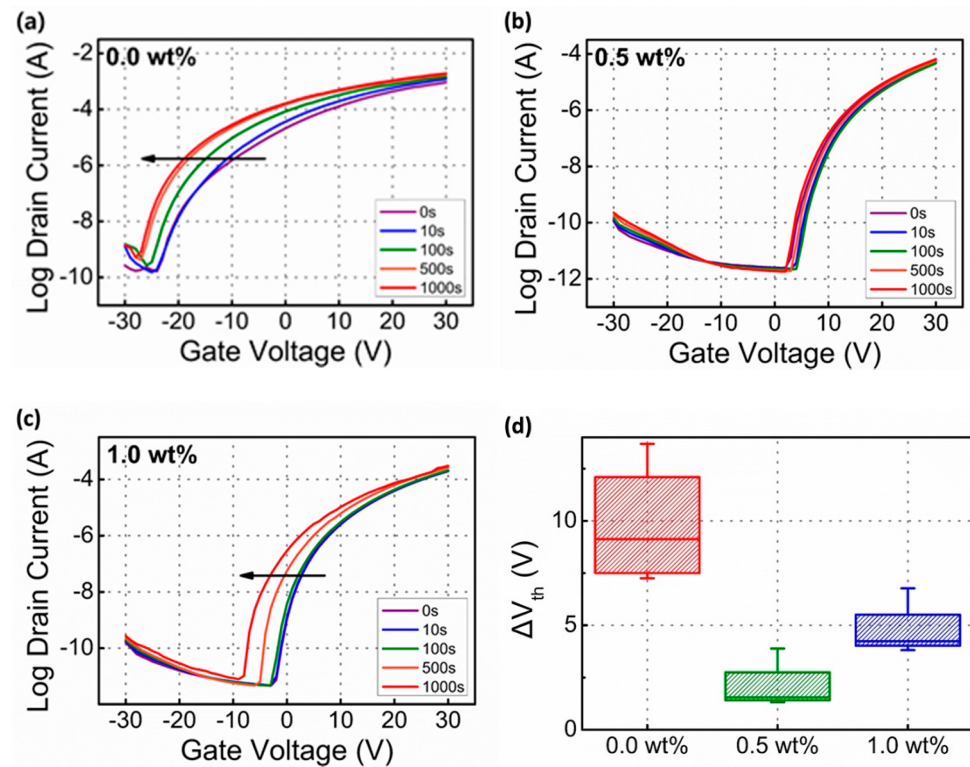


Figure 5. (a–c) Variation in transfer curves and (d) change in V_{th} of Li-doped SnO_2 based TFTs during NBS test.

4. Conclusions

In this study, sol-gel-processed Li-doped SnO_2 -based TFTs were fabricated on SiO_2/p^+ Si substrates. The influence of the Li dopant amount on the structural, chemical, optical, and electrical characteristics of the fabricated Li-doped SnO_2 -based TFTs was investigated. By adding 0.5-wt% Li, the oxygen vacancy formation process was successfully suppressed. The TFTs fabricated using 0.5-wt% Li-doped SnO_2 semiconductor films exhibited FEM in the saturation regime of $2.0 \text{ cm}^2/\text{Vs}$ and showed the enhancement-mode operation. The decreased O_{Vacancy} inside the SnO_2 -based TFTs fabricated using 0.5-wt% Li improved the negative bias stability of TFTs.

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Data Availability Statement: Data available in a publicly accessible repository.

Conflicts of Interest: The authors declare no conflict of interest.

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