Transparent thin film transistors of polycrystalline SnO$_{2-x}$ and epitaxial SnO$_{2-x}$

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Transparent thin film transistors of polycrystalline SnO$_{2-x}$ and epitaxial SnO$_{2-x}$

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ABSTRACT

We report on transparent thin film field effect transistors (TFTs) based on polycrystalline SnO$_{2-x}$ and epitaxial SnO$_{2-x}$. Polycrystalline SnO$_{2-x}$ TFTs of the top and the bottom gate geometries exhibited high mobility values of 145.7 cm$^2$/V s and 160.0 cm$^2$/V s, respectively. However, our polycrystalline SnO$_{2-x}$ devices showed non-ideal behaviors in their output and transfer characteristics; a large hysteresis was observed along with large voltage dependence. The probable origin of these non-ideal behaviors is the barrier formation across grain boundaries of polycrystalline SnO$_2$. To confirm this, we used SnO$_{2-x}$ epitaxially grown on r-plane sapphire substrates as a channel layer and compared their performance with those of polycrystalline SnO$_{2-x}$ based TFTs. Although the mobility of epitaxial SnO$_{2-x}$ TFTs was not as high as that of the polycrystalline SnO$_{2-x}$ TFTs, the non-ideal voltage dependence in output and transfer characteristics disappeared. We believe our direct experimental comparison clearly demonstrates the grain boundary issue in polycrystalline SnO$_{2-x}$.

SnO$_2$ is a transparent semiconductor with a wide bandgap (~3.6 eV). Although it becomes more difficult to dope as the bandgap becomes wider, SnO$_2$ is known to be able to have a carrier density larger than 10$^{20}$ cm$^{-3}$ and its conductance high enough to be used as a transparent conductive oxide (TCO). Examples are Sb-doped SnO$_2$, F-doped SnO$_2$, and Ta-doped SnO$_2$. SnO$_{2-x}$ is also attractive as a transparent oxide semiconductor (TOS). In this case, the oxygen vacancies act as shallow donor levels and can be controlled with an oxygen atmosphere during or after the growth. A SnO$_{2-x}$ single crystal was reported to exhibit a mobility value as high as 240 cm$^2$/V s with a carrier density of $1.3 \times 10^{17}$ cm$^{-3}$. Recently, a SnO$_{2-x}$ epitaxial film with 106 cm$^2$/V s mobility and $3.0 \times 10^{18}$ cm$^{-3}$ carrier density was demonstrated by pulsed laser ablation.

TOS is becoming more important for the development of the transparent optoelectronic device. For the application of TOS as an optoelectronic device, the demonstration of transparent thin film transistors with excellent electrical performances on the glass and/or flexible substrates is most needed. This is one of the areas that cannot be covered by conventional Si or III–V semiconductors. SnO$_2$ is one of the promising TOS candidates because of its transparency, electrical transport properties, and chemical stability. The primary conduction band in SnO$_2$ originates from the Sn 5s orbital. The large overlap of such 5s orbitals makes it possible for SnO$_2$ to exhibit good electrical properties regardless of its crystallinity.

There have been several reports on thin film transistors based on SnO$_2$ with diverse gate dielectrics: Sb-doped SnO$_2$/SiO$_2$, SnO$_2$/Al$_2$O$_3$, SnO$_2$/ZrO$_2$, SnO$_2$/HfO$_2$, SnO$_2$/SiO$_2$–Al$_2$O$_3$–HfO$_2$, and SnO$_2$/HfO$_2$. All these reports used a bottom gate structure and reported high mobility values between 96 cm$^2$/V s and 279 cm$^2$/V s, except for our previous report, which used a top gate structure with its mobility value about 20 cm$^2$/V s. Here, we demonstrate the high mobility SnO$_{2-x}$ thin film field effect transistors (TFTs) with both top and bottom gate geometries and concur with the grain boundary issue in polycrystalline SnO$_{2-x}$ by comparing the polycrystalline and epitaxial SnO$_{2-x}$ TFTs.

Here, we used oxygen vacancies as the charge carriers in the channel layer. To distinguish the insulating SnO$_2$ grown in high oxygen pressure from the intentionally oxygen vacancy doped conducting SnO$_2$ grown in low oxygen pressure, we notated SnO$_2$ for the insulating SnO$_2$ and SnO$_{2-x}$ for the conducting SnO$_2$. For polycrystalline SnO$_{2-x}$ on the glass, we used the reactive sputtering method at room temperature. A Sn metal target with 99.999% purity was used as the source material for reactive sputtering. The composition

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Figure 1(d) shows the deposited Sn-doped In$_2$O$_3$ layer was deposited in 200 mTorr oxygen pressure. An epitaxial SnO$_2$ layer was deposited in 200 mTorr oxygen pressure. In order to reduce the effect on the channel layer by the dislocations and the anti-phase boundaries, an insulating SnO$_2$ buffer layer was deposited in 200 mTorr oxygen pressure. An epitaxial SnO$_{2-x}$ thin film was deposited at 50 mTorr oxygen pressure. On the source, drain, and gate electrodes of thin film transistors, we deposited Sn-doped In$_2$O$_3$ (ITO) by laser ablation at 150 °C with stainless steel masks. A Si stencil mask was used for the channel layer.

Figure 1(a) demonstrates the transparency of the polycrystalline SnO$_{2-x}$ films on glass substrates. Figure 1(b) is a transmission electron microscopy (TEM) image and shows that the annealed SnO$_{2-x}$ film, which was deposited by the reactive sputtering method, is polycrystalline. We also performed an x-ray diffraction (XRD) measurement to confirm the phase of the annealed polycrystalline film [Fig. 1(c)]. The 0–2θ diffraction pattern of 300 nm thick polycrystalline SnO$_{2-x}$ film on a glass shows that the peaks are corresponding to SnO$_2$ (110), (101), and (211). Using the Van der Pauw Hall measurements, the electric transport properties of the 20 nm thick polycrystalline SnO$_{2-x}$ film were obtained as follows: the mobility 6.6 cm$^2$/V·s and the n-type carrier density $6.7 \times 10^{18}$ cm$^{-3}$. Figure 1(d) shows the 0–2θ diffraction pattern of SnO$_2$ grown at 800 °C on r-plane sapphire substrates by PLD. Only (101) and (202) peaks of the SnO$_2$ are observed. The detailed electrical properties of epitaxial SnO$_{2-x}$ were reported in our previous study.

It is known that tin oxide has two stable phases: SnO and SnO$_2$. SnO has a square pyramid crystal structure and 2.8 eV bandgap. On the other hand, SnO$_2$ has a rutile crystal structure and 3.6 eV wide bandgap. SnO$_{1+x}$ is a p-type material, unlike the n-type semiconductor SnO$_{2-x}$. From the XRD analysis and the Hall measurements, we were certain that our SnO$_{2-x}$ films are in the SnO$_2$ phase.

Based on the film study, we fabricated the polycrystalline SnO$_{2-x}$ TFTs in both the top and the bottom gate geometries with the HfO$_2$ gate dielectric. Figures 2(a), 2(b), 3(a), and 3(b) show the schematic cross-sectional diagrams and the top view microscopy images for the polycrystalline top and the bottom gate TFTs, respectively. The 83 nm thick HfO$_2$ was grown by the atomic layer deposition (ALD) method using Hf[N(CH$_3$)$_2$]$_4$ and H$_2$O sources at 250 °C. Due to hydrogen absorption during the ALD process, channel layer conductance usually increased by about one order after the HfO$_2$ ALD growth. We reannealed the device at 400 °C for an hour with flowing oxygen gas in the atmosphere to get rid of the hydrogens, after which the conductance of the channel usually returns to its original value.

In order to check the dielectric properties of HfO$_2$, we made MIM (metal-insulator-metal) capacitors with ITO electrodes. The capacitance–frequency (C–F) and current–voltage (I–V) characteristics were measured through Keithley 4200 SCS. After the post-ALD annealing process, the dielectric constant of HfO$_2$ was calculated to be 24.0 from the measured capacitance per unit area $(2.5 \times 10^{-7}$ F/cm$^2$), and the breakdown field was 2 MV/cm.
This dielectric constant value is consistent with other previous works.\textsuperscript{15,17,18} During the post-ALD annealing at 400°C, HfO\textsubscript{2} becomes polycrystalline [Fig. 1(b)].

Figures 2(c) and 3(c) exhibit output characteristics (I\textsubscript{DS} vs V\textsubscript{DS}) for the top and the bottom gate TFTs based on polycrystalline SnO\textsubscript{2-x}. We measured the source–drain current (I\textsubscript{DS}) by increasing source–drain voltage (V\textsubscript{DS}) at each gate–source voltage (V\textsubscript{GS}). The applied V\textsubscript{GS} varied from 3 V to −2 V in 1 V steps. The output characteristics confirm that both types of polycrystalline SnO\textsubscript{2-x} TFTs operate in an n-type depletion mode. We also measured the...
transfer curves, namely the source–drain current (I_{DS}), while varying the gate–source voltage (V_{GS}) at V_{DS} = 1 V [Figs. 2(d) and 3(d)]. Field effect transistor performances, such as the field effect mobility (\(\mu_{FE}\)), I_{on}/I_{off} ratio, and the subthreshold swing (S) were obtained by using the following general relations:

\[
\mu_{FE} = \frac{\partial I_{DS}}{\partial V_{GS}} \cdot \frac{L}{W V_{DS} C_{ox}},
\]

where L, W, and C_{ox} are the channel length (100 \(\mu\text{m}\)), the channel width, and the capacitance per unit area of HfO\(_2\) gate dielectric, respectively,

\[
\frac{I_{on}}{I_{off}} = \frac{\text{the highest current value}}{\text{the lowest current value}},
\]

\[
S = \frac{1}{\left(\frac{\partial \ln I_{DS}}{\partial V_{GS}}\right)},
\]

The maximum \(\mu_{FE}\) of the top and the bottom gate polycrystalline TFTs are 145.7 cm\(^2\)/V s and 160.0 cm\(^2\)/V s, respectively, in the linear region (V_{DS} = 1 V). The I_{on}/I_{off} values are 8.9 \times 10^7 and 3.1 \times 10^5, respectively. The subthreshold swing (S) values are 0.2 V/dec and 0.7 V/dec, respectively. The SnO\(_{2-x}\) TFTs show higher mobility when the carriers are accumulated by field effect than that of the films, probably due to the fewer oxygen vacancies in the TFT channel layer. The other is related to the gate oxide layer. During the fabrication process, the HfO\(_2\) gate dielectric goes through more diverse environments in the case of the bottom gate geometry, which may cause relatively higher leakage current (I_{GS}), leading to less ideal characteristics.

Figure 4 shows the aging effects and voltage (V_{DS}) dependence in polycrystalline SnO\(_{2-x}\) TFTs. For 2 months, the devices were kept under ambient conditions. The top gate TFT in Fig. 4(a) seems nearly not affected by aging; the field effect mobility and the I_{on}/I_{off} ratio were slightly increased and the subthreshold swing (S) remained nearly the same. However, in the case of the bottom gate TFT in Fig. 4(b), the on-state current and mobility values have decreased in 2 months, showing a larger aging effect than the top gate TFTs. Since the SnO\(_{2-x}\) channel layer was exposed, various molecules might be more easily absorbed in the channel, possibly causing the on-state conductance (I_{ON}) and the mobility decrease. The polycrystalline top gate TFT characteristics endure the aging process much better because the HfO\(_2\) gate dielectric plays an additional role of passivation. However, the polycrystalline TFTs of both the top and the bottom geometries exhibited the voltage-dependent behaviors. Figure 4(c) shows the top gate polycrystalline SnO\(_{2-x}\) TFT aging effect and V_{DS} dependence simultaneously. To directly compare the aging effect and V_{DS} dependence within one device, we had to measure the device performances right after fabrication and after passing enough time. After applying the saturation voltage V_{DS} = 5 V [Fig. 4(c) green line], the on-state current significantly decreased and the hysteresis direction switched from the counter-clockwise to the clockwise direction. We often
observed switching of the hysteresis direction after applying high $V_{DS}$, suggesting that the current paths through the grain boundaries are sensitive to the current flowing history. Figure 4(c) shows the on-state current from the as-fabricated state (black dotted line), after 5 months (red dotted line), and after applying the saturation $V_{DS}$ (blue dotted line). As the current decreased, the field effect mobility decreased after 5 months from 145.7 cm$^2$/V s to 72.8 cm$^2$/V s and further decreased to 45.6 cm$^2$/V s after applying the saturation voltage ($V_{DS} = 5$ V).

The counter-clockwise hysteresis in transfer characteristics, Figs. 2(d) and 3(d), is in contrast to the field effect transistors based on a La-doped BaSnO$_3$ channel with the same HfO$_2$ gate dielectric in which we recently reported a clockwise hysteresis in the $I_{DS}$–$V_{GS}$ curve.$^{19}$ Clockwise hysteresis (for the n-type device) is known to be caused by trap states near the interface between the channel layer and the gate dielectric.$^{18,21}$ These trap states reduce the effective electric field by $V_{GS}$, causing clockwise hysteresis. On the other hand, the counter-clockwise hysteresis usually occurs because of the dielectric property or defects inside the gate oxide, which try to maintain the electric field even when the $V_{GS}$ is reduced. There are usually three possible explanations for counter-clockwise hysteresis: (1) ferroelectricity of the dielectric, (2) mobile ions in the dielectric, and (3) charge injection and trapping inside the dielectric.$^{22,23}$ The first explanation can be ruled out because the polarization in HfO$_2$ is reported to occur usually in cubic, tetragonal, or orthorhombic phases.$^{24,25}$ These phases are usually formed when HfO$_2$ is doped with other materials$^{22,23}$ or under particular conditions.$^{26,27}$ We also exclude the second possibility since we performed the same post-ALD annealing for hydrogen elimination for both the polycrystalline SnO$_2$-x and La-doped BaSnO$_3$ TFTs.$^{18}$ Charge injection and trapping usually accompany with an increase in leakage current as increasing $V_{GS}$ due to the tunneling process is assisted by defects.$^{24,25}$ However, our polycrystalline TFTs showed very low leakage current $I_{GS}$ when compared with $I_{DS}$. Therefore, the third possibility is also a remote possibility for the counter-clockwise hysteresis in the transfer characteristics.

Both types of polycrystalline TFTs showed not only $V_{DS}$ history dependency in transfer characteristics, but also non-ideality in output characteristics and counter-clockwise hysteresis in transfer characteristics. As shown in Fig. 5, the polycrystalline TFTs exhibited not a saturation of the current but a decrease of the current after the pinch-off in low $V_{GS}$, as indicated by red arrows in Fig. 5. These cannot be explained by the negative differential resistance caused by self-heating in other studies.$^{28,29}$ Our polycrystalline TFTs showed the current decrease only in the low $V_{GS}$, whereas the self-heating should become more severe as the current increases due to the increasing $V_{GS}$.

SnO$_2$ has been used for applications such as catalysis and solid-state gas sensing, other than transparent oxide semiconductors (TOS) and transparent conductive oxides (TCO). These applications employ the surface and interface properties of SnO$_2$. It is believed that the gas response is caused not by the lattice oxygen concentration but by the chemical adsorption of oxygen and other molecules. An adsorbed molecule at the surface makes electrons transfer between SnO$_2$ and the adsorbed molecule. This has a net charge at the surface and leads to the electric field. This electric field results in band bending. For example, O$^{2-}$, a negative charge makes the band bend upward and reduce the charge carrier concentration. This leads to the formation of the depletion region at the surface. For polycrystalline SnO$_2$, this mechanism occurs at every grain boundaries and forms a double Schottky barrier between grains.$^{27}$ The grain size, the charge trap density, and the carrier density, which vary depending on the deposition conditions, seem to determine the barrier height. Such barriers are known to be related to the voltage dependence and non-ohmic behavior of the SnO$_2$ due to charge trapping in or near the grain boundaries; applying bias can be used to control the barrier height and current flow. The non-ohmic behavior is known to depend on SnO$_2$ itself, not on the electrode material.$^{4,16,21,23}$ Using such a process to modify the barrier height in the current path, the SnO$_2$ for gas sensor changes the resistance in the gas environment. These phenomena exist not only in polycrystalline SnO$_2$, but also have been reported in polycrystalline ZnO TFTs for gas sensing or varistor applications.$^{30,31}$

When comparing Figs. 4 and 5, the current values ($I_{DS}$) seem inconsistent at $V_{DS} = 1$ V and $V_{GS} = 0$ V. The $I_{DS}$ in Fig. 5 is larger than the sweep up ($V_{GS}$ varies from minus to plus bias) current in Fig. 4 and a little smaller than the sweep down ($V_{GS}$ varies from plus to minus bias) current. It is because of the residual effects from the charge traps at the grain boundaries. Due to the HfO$_2$ layer, which works as the gate oxide and a passivation layer,$^{31}$ the top gate structure polycrystalline TFT seems not much affected by the gas environment (Fig. 4). However, it shows a large current decrease by voltage, indicating that the charge trapping and untrapping in grain boundaries are modifying the barrier height. To understand and control such surface sensitive behaviors of the polycrystalline SnO$_2$ is a complicated and difficult task because the polycrystalline SnO$_2$ is composed of many grains of several orientations such as (110), (101), and (211).
In order to confirm such grain boundary related issues, we used epitaxial SnO$_2$ instead of polycrystalline SnO$_2$. Based on our previous study on epitaxial SnO$_{2-x}$ films, we fabricated the epitaxial SnO$_{2-x}$ TFT shown in Figs. 6 and 7. Because of the dislocations in epitaxial SnO$_{2-x}$ on sapphire, we used an 100 nm thick insulating buffer layer, which was fully oxidized during its growth in 200 mTorr oxygen pressure. In order to create n-type carriers in the SnO$_{2-x}$ film coming from oxygen vacancies, we used a reduced oxygen pressure of 50 mTorr for the 20 nm thick channel layer. More complete crystal structures (XRD, TEM) and electrical properties (Hall measurement) are shown in our previous study on epitaxial SnO$_{2-x}$.

The epitaxial devices showed an ideal saturation behavior of an n-type depletion mode in their output characteristics, shown in Fig. 6(c). As shown in Figs. 6(d) and 7, the field effect mobility, $I_{ON}/I_{OFF}$ ratio, and subthreshold swing (S) values were 20.5 cm$^2$/V s, $2.3 \times 10^6$, and 0.6 V/dec, respectively. The hysteresis curve in the transfer characteristics was found to be clockwise, a usual behavior coming from the defects near the interface with the HfO$_2$ gate oxide. Although the field effect mobility was not as high as that of the polycrystalline SnO$_{2-x}$ TFTs, there was no decrease in the $I_{ON}$ current or change in the hysteresis direction after applying various $V_{DS}$, although a shift of the threshold voltage has been observed. Following the previous studies, the existence of a high density threading dislocations and anti-phase boundaries in epitaxial SnO$_2$ could explain for the lower mobility in the epitaxial TFTs. In particular, on the r-plane sapphire substrates four SnO$_2$ lattice spacings and one anti-phase boundary repeat in every 2.57 nm. On the other hand, the grain size in our polycrystalline SnO$_2$ is

FIG. 6. Epitaxial SnO$_{2-x}$ TFT. (a) Cross-sectional schematic diagram of the epitaxial SnO$_{2-x}$ TFT. (b) Top view optical microscopy image. (c) Output characteristics ($I_{DS}$ vs $V_{DS}$). (d) Transfer characteristics ($I_{DS}$ vs $V_{GS}$ at $V_{DS} = 1$ V): the field effect mobility, $I_{ON}/I_{OFF}$ ratio, and the subthreshold swing (S) are 20.5 cm$^2$/V s, $2.3 \times 10^6$, and 0.6 V/dec, respectively. It exhibits clockwise hysteresis.

FIG. 7. Voltage ($V_{DS}$) dependence measurement of epitaxial SnO$_{2-x}$ TFT. The on current level and clockwise hysteresis are not affected by changing $V_{GS}$. 

1st, $V_{DS} = 1$ V
2nd, $V_{DS} = 5$ V
3rd, $V_{DS} = 1$ V
approximately 7–10 nm from the XRD and the TEM data. Although the crystalline defects in epitaxial SnO$_2$–x do not seem to affect the stability, they seem to be the cause for the lower mobility in epitaxial SnO$_2$. In polycrystalline SnO$_2$–x, grain boundaries seem responsible for the instability, such as hysteretic behavior and voltage dependence, but its effect on mobility seems minimal. This work confirms that the grain boundaries in polycrystalline SnO$_2$–x are the origin for the voltage-dependent hysteretic behavior. If the polycrystalline SnO$_2$–x TFT is used, it may be possible to demonstrate epitaxial SnO$_2$–x TFTs with mobility as high as those of the polycrystalline SnO$_2$–x TFTs without the electrical instability.

In summary, we demonstrated transparent thin film transistors of polycrystalline SnO$_2$–x and epitaxial SnO$_2$–x with HfO$_2$ gate dielectric. The polycrystalline SnO$_2$–x TFTs were fabricated in both the top and the bottom gate structures, showing hysteretic and non-ideal behaviors in the transfer and the output characteristics, despite the high mobility values. No such non-ideal behavior was observed for the epitaxial SnO$_2$–x TFT, suggesting that the grain boundaries in SnO$_2$–x are the origin of such a behavior. The use of polycrystalline SnO$_2$–x TFTs will require thorough understanding of grain boundaries in SnO$_2$–x.

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