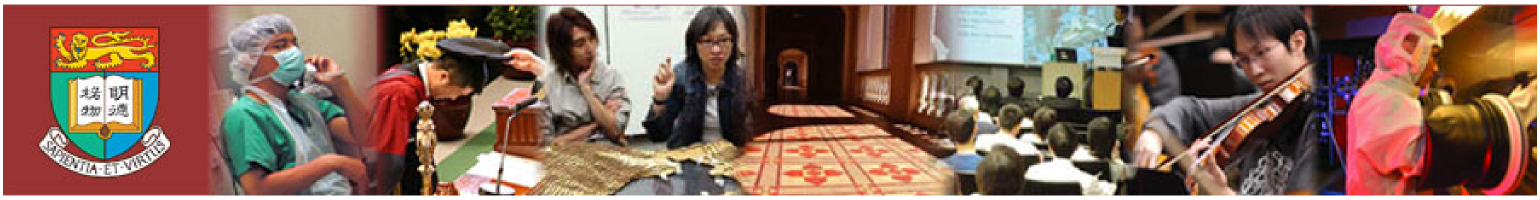

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Author(s)	**Deng, LF; Liu, YR; Choi, HW; Xu, JP; Che, CM; Lai, PT**
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Effects of Annealing Temperature and Gas on Pentacene OTFTs With HfLaO as Gate Dielectric

L. F. Deng, Y. R. Liu, H. W. Choi, *Senior Member, IEEE*, J. P. Xu, C. M. Che, and P. T. Lai, *Senior Member, IEEE*

***Abstract*—Pentacene organic thin-film transistors (OTFTs) with high-κ HfLaO as gate insulator were fabricated. HfLaO film was prepared by sputtering method. To improve the film quality, the dielectric was annealed in N_2, NH_3, or O_2 at two temperatures, i.e., 200 °C and 400 °C, respectively. The I–V characteristics of the OTFTs and C–V characteristics of corresponding organic capacitors were measured. The OTFTs could operate at a low operating voltage of below 5 V, and the dielectric constant of the HfLaO film could be above ten. For all the annealing gases, the OTFTs annealed at 400 °C achieved higher carrier mobility than their counterparts annealed at 200 °C (with the one annealed in NH_3 at 400 °C showing the highest carrier mobility of 0.45 $cm^2/V \cdot s$), which could be supported by SEM images which indicate that pentacene tended to form larger grains on HfLaO annealed at 400 °C than on that annealed at 200 °C. The C–V measurement of the organic capacitors indicated that the localized charge density in the organic semiconductor/oxide was lower for the 400 °C annealing than for the 200 °C annealing. Furthermore, through the characterization of gate current leakage, HfLaO film annealed at 400 °C achieved much smaller leakage than that annealed at 200 °C. Since the maximum processing temperature of ITO glass substrates is around 400 °C, this study shows that 400 °C is suitable for the annealing of HfLaO film in high-performance OTFTs on glass substrate.**

***Index Terms*—Dielectric, HfLaO, high κ, organic thin-film transistor (OTFT).**

I. INTRODUCTION

ORGANIC thin-film transistors (OTFTs) have emerged as an important electronic device since the electrical conductivity of organic material was discovered [1]–[3]. In comparison with their inorganic counterparts, organic devices are advantageous in many aspects. First of all, they can be easily manufactured in large area, and so, this makes them particularly suitable for the application of displays [4]. Moreover, the fabrication temperatures of organic devices are usually much lower than those of inorganic devices, and hence, the former can be realized on flexible plastic substrate. Furthermore, organic products are often light. The advantages mentioned previously are helpful for organic devices to be applied in portable equipment. Moreover, lithography is usually not needed for the fabrication of OTFTs. Consequently, it costs less to fabricate organic devices than inorganic devices. In a word, OTFTs have high potential to be applied in large-area flexible display [5], [6], radio-frequency identification card [7], electronic paper, sensors [8]–[10], and so on.

Carrier mobility is a significant parameter to evaluate the performance of OTFTs, because it decisively influences the speed of the device and relevant circuit. Pentacene OTFTs based on silicon dioxide gate dielectric prepared by oxidation have a mobility comparable to that of amorphous silicon thin-film transistors [11]. However, the operating voltage of the OTFTs is usually too high, and typical value is often larger than 15 V. The high operating voltage leads to excessive power consumption, as well as complicated power supply. As a result, organic devices add extra cost and also are not suitable for low-voltage portable equipment. The high operating voltage of OTFTs originates from the small capacitance of their gate dielectric. A straightforward way to solve this problem is to scale down the thickness of the gate insulator. However, too thin insulator may cause large gate current leakage. An alternative is to replace the gate insulator of low dielectric constant with material of high dielectric constant (high κ). Dimitrakopoulos *et al.* [12], [13] pioneered in 1999 the use of high-κ oxides as the gate dielectric of OTFTs. By this way, the operating voltage of OTFTs was reduced to as low as 5 V. Several dielectric films were synthesized through sputtering method. There were barium zirconate titanate (BZT; $\kappa = 17.3$), barium strontium titanate (BST; $\kappa = 16$), and Si_3N_4 $(\kappa = 6.2)$. Pentacene OTFTs based on these dielectrics had a carrier mobility higher than 0.3 and 0.5 $cm^2/V \cdot s$ for the BZT and BST dielectrics, respectively, while for the Si_3N_4 case, the mobility of the pentacene OTFT could amount to 0.6 $cm^2/V \cdot s$. Kang *et al.* in 2004 reported pentacene OTFTs using Gd_2O_3 as the gate insulator [14], with the Gd_2O_3 film grown by ion-beam-assisted deposition. The operating voltage of the OTFTs was

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L. F. Deng, H. W. Choi, and P. T. Lai are with the Department of Electrical and Electronic Engineering, The University of Hong Kong, Pokfulam, Hong Kong (e-mail: laip@eee.hku.hk).

Y. R. Liu is with the School of Electronic and Information Engineering, South China University of Technology, Guangzhou 510640, China.

J. P. Xu is with the Department of Electronic Science and Technology, Huazhong University of Science and Technology, Wuhan 430074, China.

C. M. Che is with the Department of Chemistry, The University of Hong Kong, Pokfulam, Hong Kong.

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10–20 V, and the mobility was 0.1 $\mathrm{cm}^2/\mathrm{V} \cdot \mathrm{s}$. In 2003, Lee *et al.* [15] reported pentacene OTFTs based on Al_2O_3, which was deposited by magnetron radio-frequency sputtering. The mobility was 0.14 $\mathrm{cm}^2/\mathrm{V} \cdot \mathrm{s}$, and the device could operate at a voltage of 30 V. Hf-based dielectric applied in OTFTs was first reported by Tardy *et al.* in 2007 [16]. Pentacene-based OTFT with HfO_2 ($\kappa = 22$ for anodic oxidation and $\kappa = 11$ for sol-gel) gate dielectric was grown by anodic oxidation method and sol-gel method, respectively. The maximum mobility was around 0.12 $\mathrm{cm}^2/\mathrm{V} \cdot \mathrm{s}$, and the device could operate at about 2.5 V. HfLaO is a promising gate dielectric to be applied in inorganic MOSFETs because Fermi-energy pinning could be reduced by adding lanthanum (La) into hafnium oxide [17], [18]. Also, OTFTs with HfLaO gate dielectric had been reported and demonstrated high performance [19]–[22].

The carrier transport of OTFTs occurs at the interface between the organic semiconductor film and the gate dielectric film. Therefore, treatment of the gate dielectric film can significantly affect the performance of the devices [23]. In the case of OTFTs with HfLaO gate dielectric, the effects of annealing temperature, together with annealing gas, on their performance have not been reported. This is a major concern for devices fabricated on glass substrate or plastic substrate, because there usually exists a tradeoff between device performance and maximum processing temperature. Therefore, this study concentrates on how the annealing temperature of the gate dielectric affects the quality of HfLaO film itself and the performance of relevant OTFTs.

II. Experimental Details

First, n-type silicon substrate was treated according to the standard RCA method to achieve clean surface. The silicon was $\langle 100 \rangle$ type, and its resistivity was $0.5-0.7\ \Omega \cdot \mathrm{cm}$. Next, the samples were kept in hydrofluoric acid (concentration of 5%) for 1 min to remove the native oxide and then washed up by deionized water. Then, the wafer was inserted into the chamber of a sputterer (Denton Vacuum LLC Discovery 635) to deposit a layer of HfLaO film. The target was HfLa alloy with an atomic ratio of 6:4 for Hf and La. Radio-frequency sputtering mode was used at a power of 35 W and a pressure of 2.0×10^{-6} torr. During the sputtering process, argon and oxygen were injected into the chamber with gas flow rates of 24 and 6 sccm, respectively. After sputtering, the samples were annealed in N_2, NH_3, or O_2 to improve the surface and bulk qualities of the HfLaO dielectric. To study the effects of annealing temperature on the performance of the devices, 400 °C and 200 °C were chosen for all the annealing gases because they are roughly the upper limit for the processing temperature of ITO glass and plastic substrates, respectively. For all the aforementioned combinations, the annealing time was 10 min, and the gas flow rate was 1000 mL/min. After the annealing, hydrofluoric acid with a concentration of 20% was used to remove the back oxide on the substrate. Then, the samples were put into the chamber of an evaporator Edwards Auto 306, and a layer of pentacene (purchased from Sigma-Aldrich) was evaporated on the dielectric at a vacuum of 4.0×10^{-6} torr through sublimation. The growth rate of pentacene film was 1.2 nm/min, which was monitored by a quartz-crystal oscillator. The thickness of pentacene film was 30 nm. Finally, gold drain and source electrodes were formed through a shadow mask at a vacuum of 8.0×10^{-6} torr. The channel length and channel width of the devices were measured by a digital microscope, and the values were 30 and 200 μm on the mask, respectively.

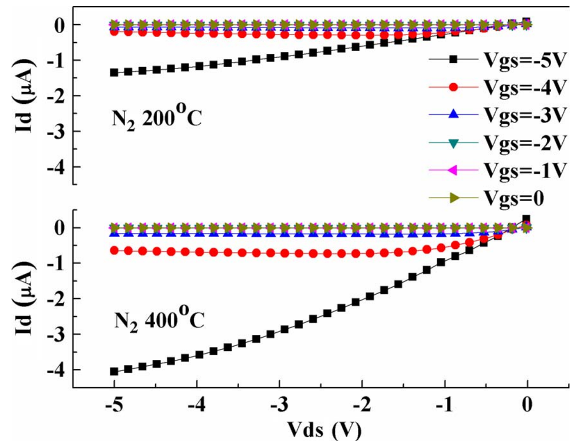


Fig. 1. Output characteristic comparison of OTFTs annealed in N_2.

To characterize the quality of the HfLaO dielectric, dummy wafer was prepared. Aluminum was evaporated on the dielectric at a vacuum of 8.0×10^{-6} torr. Silicon MOS capacitors (Si–HfLaO–Al) were fabricated by lithography method. Organic MOS capacitors are on the same wafer of OTFTs with a structure of Au–pentacene–HfLaO–Si.

An HP 4145B semiconductor parameter analyzer was used to measure the output characteristics and transfer characteristics of the OTFTs and the I–V characteristics of the silicon MOS capacitors. C–V characteristics of the silicon MOS capacitors and the organic MOS capacitors (Au–pentacene–HfLaO–Si) were characterized by HP 4284A Precision LCR Meter. All the electrical measurements were conducted in a shielded probe station. The thickness of HfLaO film was gotten by an ellipsometer Wvase 32 made by J. A. Woollam Co., Inc. Morphology of the pentacene film was obtained by a scanning electron microscope.

III. Results and Discussions

Figs. 1–3 show the output characteristics of the six OTFTs with gate dielectric annealed in N_2, NH_3, or O_2 at 200 °C and 400 °C, respectively. According to these graphs, all the OTFTs can operate with a supply voltage below 5 V and display good field-effect characteristics. The drive current for the OTFTs exceeded 1 μA. Among them, OTFT annealed in NH_3 at 400 °C achieves the largest drive current of 5 μA, while OTFT annealed in N_2 at 200 °C has the smallest drive current of 1.3 μA. For each annealing gas, OTFT annealed at 400 °C obtains larger drive current than that annealed at 200 °C.

Figs. 4–6 show the comparison of transfer characteristics of the OTFTs annealed at 400 °C and 200 °C, respectively. Transfer characteristics can be used to extract the major parameters of a field-effect transistor. Carrier mobility is one significant parameter for the evaluation of OTFTs, because it affects the speed of the devices. According to the current–voltage

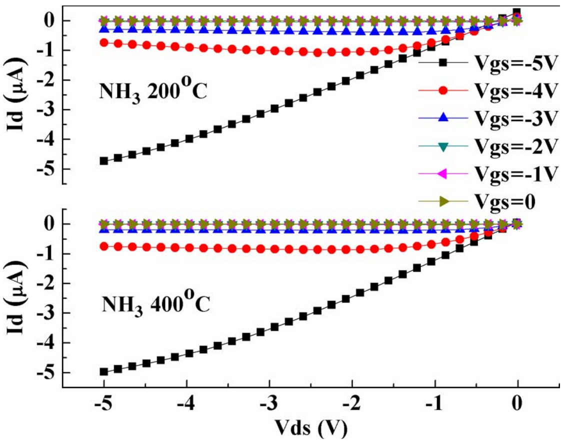


Fig. 2. Output characteristic comparison of OTFTs annealed in NH_3.

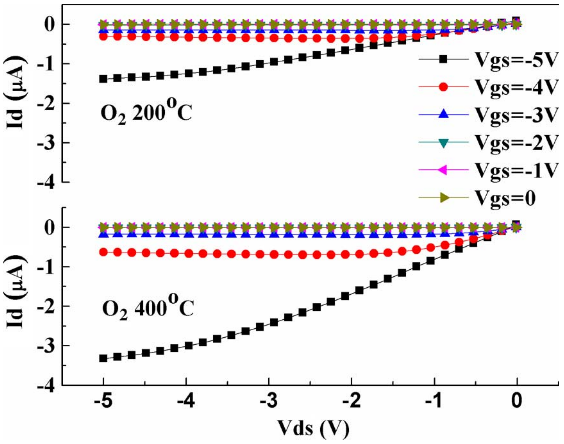


Fig. 3. Output characteristic comparison of OTFTs annealed in O_2.

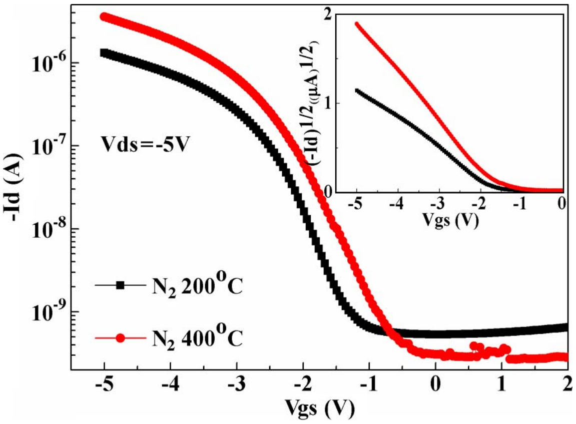


Fig. 4. Comparison of transfer characteristics of OTFTs annealed in N_2.

expression (1) of a field-effect transistor operating in the saturation region, carrier mobility can be derived as (2), where I_d is the drain current, μ is the mobility, C_{ox} is the capacitance per unit area of the gate dielectric, W is the channel width, L is the channel length, V_{gs} is the gate voltage relative to the source, and V_{th} is the threshold voltage

$$I_d = -\frac{1}{2}\mu C_{\text{ox}}\frac{W}{L}(V_{\text{gs}} - V_{\text{th}})^2 \tag{1}$$

$$\mu = \frac{2L}{WC_{\text{ox}}}\left(\frac{\partial\sqrt{-I_d}}{\partial V_{\text{gs}}}\right)^2. \tag{2}$$

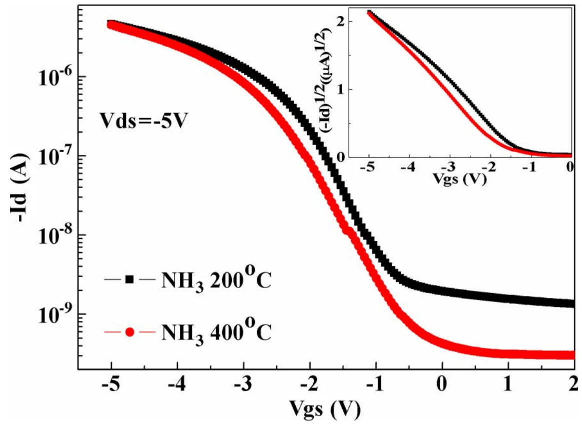


Fig. 5. Comparison of transfer characteristics of OTFTs annealed in NH_3.

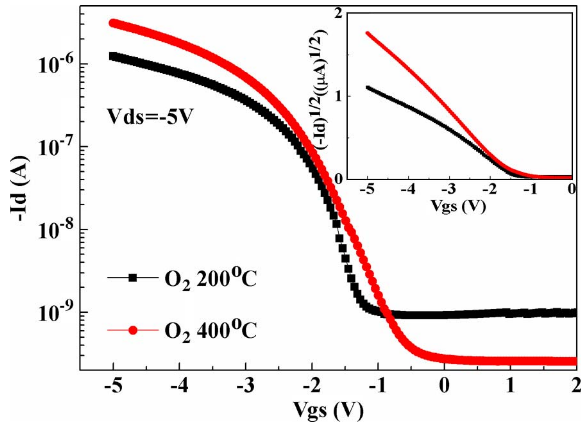


Fig. 6. Comparison of transfer characteristics of OTFTs annealed in O_2.

For each annealing gas, the carrier mobility of the OTFT annealed at 400 °C is larger than that annealed at 200 °C. Hence, the interface characteristics between the 400 °C annealed HfLaO film and pentacene are better than those of the 200 °C annealed counterpart. The OTFT annealed in NH_3 at 400 °C got the highest mobility of 0.45 $\text{cm}^2/\text{V}\cdot\text{s}$, while the OTFT annealed in O_2 at 200 °C has the lowest mobility of 0.13 $\text{cm}^2/\text{V}\cdot\text{s}$. For NH_3 annealing, nitrogen atoms are incorporated at the gate dielectric surface and can passivate the traps at the surface and, thus, trap-related carrier scattering, resulting in higher carrier mobility [23], [24].

The interface characteristics between HfLaO and pentacene can be further analyzed through the measurement of C–V characteristics of the organic MOS capacitors (Au–pentacene–HfLaO–Si units). Fig. 7 shows the C–V characteristics of the organic MOS capacitors measured at 100 kHz. According to the Mott–Schottky equation (3), the localized charge can be extracted from the capacitance variation in the depletion region [25], [26]

$$\frac{\partial(1/C^2)}{\partial V_g} = \frac{-2}{q\varepsilon_0\varepsilon_s N_A}. \tag{3}$$

In (3), C is the capacitance per unit area of the organic MOS capacitor, V_g is the voltage between the Au electrode and the substrate, q is the electron charge, ε_0 is the vacuum permittivity, N_A is the concentration of localized charge, and ε_s is the relative permittivity of pentacene (3.0 is chosen for the calculation).

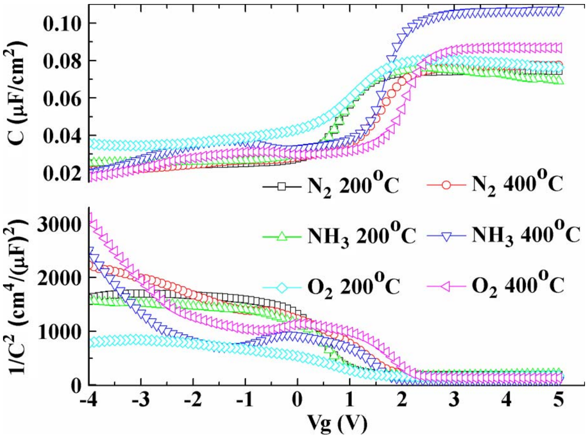
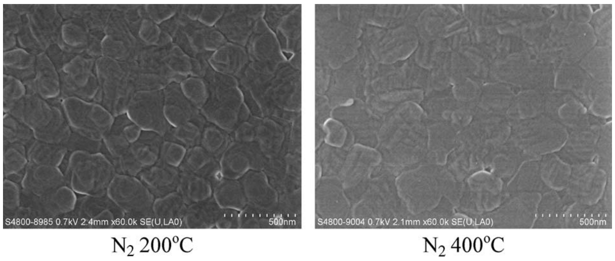
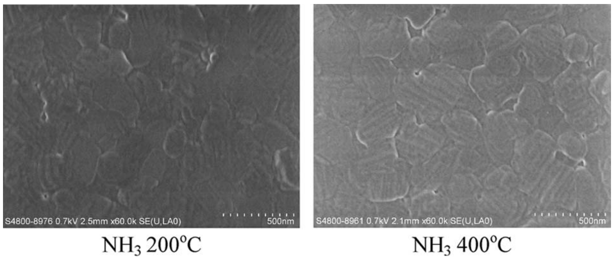


Fig. 7. Comparison of C–V characteristics of Au–pentacene–HfLaO–Si units for different annealing conditions.



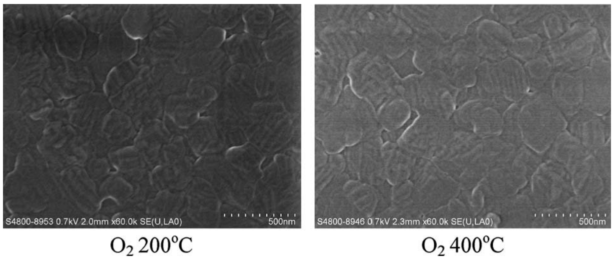
N_2 200°C N_2 400°C

Fig. 8. Comparison of pentacene morphology on HfLaO annealed in N_2.



NH_3 200°C NH_3 400°C

Fig. 9. Comparison of pentacene morphology on HfLaO annealed in NH_3.



O_2 200°C O_2 400°C

Fig. 10. Comparison of pentacene morphology on HfLaO annealed in O_2.

TABLE I
DEVICE PARAMETERS OF THE OTFTS AND CAPACITORS

Gases	N_2	N_2	NH_3	NH_3	O_2	O_2
Temperature (°C)	200	400	200	400	200	400
μ (cm^2/Vs)	0.16	0.29	0.32	0.45	0.13	0.27
Vth (V)	-1.54	-1.56	-1.10	-1.55	-1.26	-1.40
SS (V/dec)	0.502	0.601	0.579	0.628	0.337	0.519
On/off (10^3)	2.50	13.3	3.67	14.8	1.34	12.2
Idm(μA)	1.31	3.57	4.60	4.49	1.23	3.10
Cox (μF/cm^2)	0.25	0.27	0.24	0.26	0.26	0.26
Tox (nm)	35.9	34.1	35.3	34.7	36.0	34.9
κ value	10.1	10.3	9.54	10.3	10.6	10.3
N_A ($10^{17}cm^{-3}$)	8.59	1.39	8.89	0.447	4.52	0.443
J_{leak}($10^{-7}A/cm^2$)	12.9	2.98	6.45	4.17	11.6	3.20

SS: Sub-threshold swing;
On/off: on/off current ratio;
Tox: thickness of the dielectric
Idm: drive current when $V_{gs}=V_{ds}=-5V$
J_{leak}: gate dielectric current leakage when electric field at 1MV/cm

The value of N_A can be derived by calculating the slope of the $C^{-2}-V_g$ curve in the depletion region. The localized charge density in the organic MOS capacitor with HfLaO annealed at 400 °C is much lower than that for HfLaO annealed at 200 °C for all three annealing gases. In the case of NH_3 or O_2, the organic MOS capacitor annealed at 400 °C acquires a localized charge density one order lower than that at 200 °C. Carrier transport in the channel of OTFTs is influenced by the localized charge in the organic semiconductor. Less localized charge can explain why OTFTs annealed at 400 °C got higher mobility than those annealed at 200 °C for all three annealing gases.

The morphology of pentacene film on HfLaO can further help to explain why OTFTs annealed at 400 °C exhibit higher carrier mobility than those annealed at 200 °C for the annealing gas N_2, NH_3, or O_2. Figs. 8–10 show the morphology of pentacene on HfLaO dielectrics by using scanning electron microscopy. By comparing these photographs, the grains of pentacene on HfLaO annealed at 400 °C are larger than those annealed at 200 °C for the same annealing gas N_2, NH_3, or O_2. The larger the grain, the fewer the grain boundary in the organic semiconductor film, thus resulting in less scattering between the carriers and grain boundaries. Consequently, OTFTs annealed at 400 °C obtain a higher carrier mobility than OTFTs annealed at 200 °C.

Generally, the mobility of OTFTs is also influenced by the grain boundaries of the organic film in addition to the localized charge, as scattering by the defects at the grain boundaries affects the transport of carriers. The larger the grains, the lower the density of grain boundaries, thus less carrier scattering. In this paper, we concentrate on the comparison of samples annealed at 200 °C and 400 °C for the same annealing gas. In the case of annealing gas, higher annealing temperature results in lower density of localized charge and larger grain size than lower annealing temperature, hence higher mobility for the former. However, lower density of localized charge does not necessarily mean higher mobility for different annealing gases due to the other important factor—grain size.

In the case of the same annealing temperature, OTFT with the dielectric annealed in NH_3 achieves a higher carrier mobility than those annealed in N_2 or O_2 (the latter two have almost the same value). This could be explained as follows. Nitrogen and hydrogen atoms decomposed from NH_3 could passivate the surface of the HfLaO film (next to the conduction channel of the OTFT) and hence decrease the traps there [23], [24]. Consequently, trap-related carrier scattering is reduced, and the OTFTs annealed in NH_3 realize a higher carrier mobility.

For the same annealing gas (NH_3, N_2, or O_2), the samples annealed at 400 °C have an enhanced performance in comparison with their respective counterparts annealed at 200 °C. This is because the HfLaO dielectric treated at higher temperature is denser than that annealed at lower temperature. This is supported by the ellipsometry results shown in Table I.

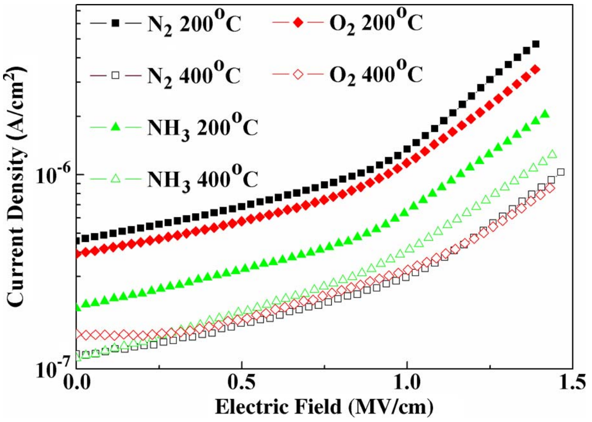


Fig. 11. Comparison of current leakage of HfLaO for different annealing conditions.

Densified dielectric film could have less defects and, thus, less traps hindering the carrier flow in the channel of the OTFTs. As a result, the OTFTs annealed at 400 °C obtain an improved performance than those annealed at 200 °C.

The enhanced quality of the HfLaO film annealed at 400 °C could be further proved by means of leakage measurement. To characterize the leakage of the dielectric, the I–V characteristics of the Si MOS capacitors (Al–HfLaO–Si) are measured. The applied voltage is divided by dielectric thickness to transform to electric field in Fig. 11. In the graphs, the dielectric annealed at 400 °C shows less leakage than that annealed at 200 °C for N_2, NH_3, or O_2 annealing. It indicates that the HfLaO film gets enhanced electrical characteristics through annealing at a higher temperature of 400 °C. This can be attributed to better densification of the dielectric film and, thus, decrease of defects. For all the films, the leakage is on the order of 10^{-7} A/cm^2, with the exception of annealing at 200 °C in N_2 or O_2, whose larger leakage is on the order of 10^{-6} A/cm^2. In the case of leakage current, defects in the dielectric film play an important role. HfLaO film annealed at 400 °C displays less leakage than that annealed at 200 °C for each gas because more thermal energy at higher temperature can remove defects more effectively. According to Fig. 11, all the three samples annealed at 400 °C have about the same leakage because, at this higher temperature, the three films can densify to achieve roughly the same bulk quality. However, at the lower temperature of 200 °C with little densification, nitrogen atoms dissociated from NH_3 can passivate the defects at the surface of the film and thus give a lower leakage for the NH_3-annealed sample as compared to the N_2- and O_2-annealed samples. Other major parameters of the OTFTs and capacitors are listed in Table I.

IV. CONCLUSION

Pentacene OTFTs with high-κ HfLaO as gate dielectric were fabricated. The dielectric films were prepared by sputtering method and annealed in N_2, NH_3, or O_2 at 200 °C and 400 °C, respectively, to improve the surface/bulk qualities of the dielectric films. The output characteristics and transfer characteristics of the OTFTs were measured. The devices could operate at a supply voltage as low as 5 V and presented good field-effect characteristics. OTFTs annealed at 400 °C displayed higher carrier mobility than those annealed at 200 °C for all the three gases. OTFT annealed in NH_3 at 400 °C obtained the highest carrier mobility of 0.45 cm^2/V · s, while that annealed in O_2 at 200 °C acquired the lowest carrier mobility of 0.13 cm^2/V · s. The C–V characteristics of the organic capacitors were measured. Organic capacitors with HfLaO annealed at 400 °C achieved less localized charges than those annealed at 200 °C. SEM images indicated that the pentacene film intended to grow larger grains on HfLaO film annealed at 400 °C than on HfLaO film annealed at 200 °C. Moreover, for all the three annealing gases, HfLaO film annealed at 400 °C exhibited smaller current leakage than that annealed at 200 °C. Since the maximum processing temperature for ITO glass substrate is around 400 °C, this study shows that 400 °C is suitable for the annealing of HfLaO gate dielectric used in high-performance OTFTs.

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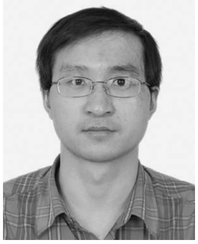
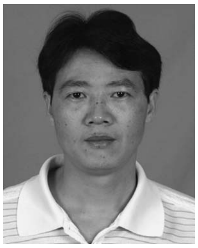


L. F. Deng received the B.S. degree in applied physics from Hefei University of Technology, Hefei, China, in 2003 and the M.Sc. degree in microelectronics from Huazhong University of Science and Technology, Wuhan, China, in 2007. He is currently working toward the Ph.D. degree in the Department of Electrical and Electronic Engineering, The University of Hong Kong, Pokfulam, Hong Kong.

His current research concentrates on organic thin-film transistors.



Y. R. Liu received the B.S. degree in semiconductor physics from Jiangxi University of Science and Technology, Ganzhou, China, in 1991, the M.Sc. degree in condensed physics from Sichuan University, Chengdu, China, in 1996, and the Ph.D. degree in microelectronics and solid-state electronics from South China University of Technology, Guangzhou, China, in 2007.

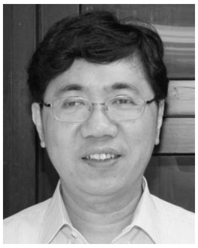
He is currently an Associate Professor with the School of Electronic and Information Engineering, South China University of Technology. His current research interests include inorganic/organic thin-film transistors, solar cells, and microsensors.

H. W. Choi (SM’09) received the Ph.D. degree in plasma processing of GaN materials from the National University of Singapore, Singapore, in 2003.

Between 2001 and 2004, he was a Postdoctoral Fellow with the Institute of Photonics, University of Strathclyde, Glasgow, U.K., where he carried out pioneering development work on GaN-based emissive micro-light-emitting-diode arrays, demonstrating applications in the areas of microdisplays and high-efficiency light sources. Since 2004, he has been with The University of Hong Kong, Pokfulam, Hong Kong, where he is currently an Associate Professor with the Department of Electrical and Electronic Engineering, with keen interest in solid-state lighting, microcavities, microdisplays, and laser micromachining.

J. P. Xu received the B.S., M.S., and Ph.D. degrees in microelectronics from Huazhong University of Science and Technology (HUST), Wuhan, China, in 1982, 1984, and 1993, respectively.

From 1997 to 1999, he was a Postdoctoral Fellow with The University of Hong Kong, Pokfulam, Hong Kong, where he was engaged in the study of thin gate dielectric for very large scale integration and gate dielectric of SiC MOS devices. He is currently a Full Professor with the Department of Electronic Science and Technology, HUST. His current research interests include the high-κ gate dielectric MOS devices, SONOS nonvolatile memory, and the reliability of small MOSFETs.



C. M. Che was born in Hong Kong in 1957. He received the Ph.D. degree from The University of Hong Kong (HKU), Pokfulam, Hong Kong, in 1982 under the supervision of Prof. Chung-Kwong Poon.

In the years 1980–1983, he was with the California Institute of Technology, Pasadena, where he worked with Prof. Harry B. Gray. He is currently with the Department of Chemistry, HKU, where he was a Lecturer in 1983 and has been a Professorial Chair since 1992. His research activities encompass diverse areas of inorganic and organic syntheses, organometallic and inorganic photochemistry, metal catalysts, luminescent materials, and inorganic medicines. He has published over 730 papers in leading chemistry journals.

Prof. Che has been an elected member with the Chinese Academy of Sciences (China) since 1995. He was a recipient of many prizes/recognitions, including the following: the First Class Prize of the State Natural Science Award from China in 2007, the TWAS Prize in Chemistry in 2006, the Seaborg Lectureship at the University of California, Berkeley, in 2007, and the Edward Clark Lee Lectureship at The University of Chicago, Chicago, IL, in 2008. He is listed as one of the highly cited researchers worldwide by www.ISIHighlyCited.com.

P. T. Lai (M’90–SM’04) received the B.Sc.(Eng.) and Ph.D. degrees from The University of Hong Kong, Pokfulam, Hong Kong. His Ph.D. research was on the design of a small-sized MOS transistor with emphasis on narrow-channel effects. The work involved the development of both analytical and numerical models, the study of this effect in relation to different isolation structures, and the development of efficient numerical algorithms for device simulation.

He was a Postdoctoral Fellow with the University of Toronto, Toronto, ON, Canada. He has proposed and implemented a novel self-aligned structure for bipolar transistors and designed and implemented an advanced polyemitter bipolar process with emphasis on self-alignment and trench isolation. He is currently a Professor with the Department of Electrical and Electronic Engineering, The University of Hong Kong. His current research interests are on thin gate dielectrics for FET devices based on Si, SiC, GaN, Ge, and organics and on microsensors for detecting gases, heat, light, and flow.