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Designing high *k* dielectric films with LiPON–Al_2O_3 hybrid structure by atomic layer deposition

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A large amount of ultra-low-power consumption electronic devices are urgently needed in the new era of the internet of things, which demand relatively low frequency response. Here, atomic layer deposition has been utilized to fabricate the ion polarization dielectric of the LiPON–Al_2O_3 hybrid structure. The LiPON thin film is periodically stacked in the Al_2O_3 matrix. This hybrid structure presents a frequency-dependent dielectric constant, of which *k* is significantly higher than the aluminum oxide matrix from 1 kHz to 200 kHz in frequency. The increased dielectric constant is attributed to the lithium ions shifting locally upon the applied electrical field, which shows an additional polarization to the Al_2O_3 matrix. This work provides a new strategy with promising potential to engineers for the dielectric constant of the gate oxide and sheds light on the application of electrolyte/dielectric hybrid structure in a variety of devices from capacitors to transistors.

Keywords: high *k* dielectric, atomic layer deposition, polarization

PACS: 77.22.Ch, 77.55.D–, 81.15.–z **DOI:** 10.1088/1674-1056/ac3736

1. Introduction

In the coming era of the internet of things (IoT), many smart terminals are urgently needing ultra-low-power consumption ($<$ 1 μW power consumption), rather than high-performance chips or extremely high operating frequencies ($<$ 100 kHz).[1–4] High dielectric constant (*k*: the ratio of the permittivity of the substance to the permittivity of the free space) materials are the core of various electronic devices. Higher *k* can offer relatively low power consumption, which is needed for many devices from capacitors to transistors.[2,5] In particular, it plays a crucial role in controlling the accumulation of mobile charge carriers at the interface upon applying external voltage. The dielectric properties greatly affect the electrical performance of devices.[2,6] High *k* gate dielectrics enable the transistor to operate at a lower voltage, and provide a relatively thicker physical thickness while maintaining the high capacitance characteristic.[5] Besides, high *k* materials can lower threshold voltage and subthreshold swing, and thus can reduce the power consumption effectively.[5]

Different kinds of high *k* dielectrics have been developed for microelectronics, including inorganic, polymer, solid electrolyte dielectrics, alkali metal ion-incorporated dielectrics, and so on.[2,3,5,7–15] Among them, ionic materials present outstanding high dielectric constant, which is frequency dependent. Their dielectric constants are mainly based on ion polarization with long-distance such as electric-double-layer (EDL) with ion-incorporated Al_2O_3-related dielectrics, or ion polarization with short-distance (locally shift), e.g., the sodium beta-alumina systems.[7,12,16–19] The *k* of these sodium beta-Al_2O_3 systems show up to $\sim$ 200 at low frequency,[16] which is far larger than that of the Al_2O_3 ($\sim$ 9) matrix. Such ionic materials have potential in IoT devices. However, the fabrication process of those thin high *k* dielectric films is mostly based on solution processes, which is not compatible with the semiconductor fabrication process.[4,7,16,17]

As for traditional high *k* materials, *k* is the intrinsic property of the materials, such as Al_2O_3 ($\sim$ 9), HfO_2 ($\sim$ 22), etc. The atomic layer deposition (ALD) is a uniquely superior gas-phase thin-film technique for deposition of uniform, conformal thin films with atomic-level precision. It is widely utilized to grow high *k* materials in the semiconductor industry.[5,20,21] ALD can deposit solid-state Li ions-based electrolyte materials.[22–25] Therefore, high *k* dielectrics (Al_2O_3, HfO_2, etc.) are expected to have potential to increase their dielectric constants through ion incorporations by ALD.

Based on this, we investigate a nano-hybrid structure deposited by ALD in this work, with Al_2O_3 film and LiPON electrolyte layers grown periodically. The *k* of this hybrid structure shows frequency-dependent characteristics. The hybrid thin film shows potential as the gate oxide for thin film transistors thanks to its higher *k* than that in the Al_2O_3 matrix. X-ray photoelectron spectroscopy (XPS) was used to characterize the composition of the hybrid structure. High resolu-

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tion transmission electron microscope (HRTEM) is utilized to characterize the thickness in the cross-sectional view. Electrical measurements are also performed to characterize the dielectric properties.

2. Experimental details

High purity nitrogen gas was used both as the carrier and purge gas for all precursors with a gas flow of 30 sccm. All substrates used in this study were split from a 2-inch n-type Si (100) single side polished wafer. The substrates were ultrasonically cleaned by using stepwise acetone, methanol, and isopropyl alcohol, blown dry by high-purity nitrogen gas (99.99%) before loading into the ALD reactor.

Five cycles of ALD of Al_2O_3 and the following two cycles of LiPON were grown on the substrate as the first full reaction cycle. Nine full reaction cycles were followed, including the sequence of ten cycles of Al_2O_3/2 cycles of LiPON. The surface of LiPON reacts with moisture, oxygen, and carbon components in air, resulting in degradation in ionic conductivity.[26,27] Therefore, for this work, another five cycles of ALD of Al_2O_3 were grown on the surface of the stack as the protective layer. This sample is labeled as 1:5 of LiPON to Al_2O_3 in ALD cycles.

In order to increase the dielectric constant, an optimized ratio between LiPON and Al_2O_3 is desired. Another two samples were fabricated with the same recipe but only the LiPON cycles were varied to be 10 cycles (1:1 to Al_2O_3 in cycle number) and 1 cycle (1:10 to Al_2O_3 in cycle number) for each repeating unit, respectively.

Lithium hexamethyldisilazide (LiHMDs) and diethyl phosphoramidate (DEPA) were used as precursors to deposited LiPON film. The ALD process was carried out with a sequence of 10 ms LiHMDs/20 s purge/30 ms DEPA/20 s purge. The Al_2O_3 film was grown by trimethylaluminum (TMA) and water vapor, consisting of a 30 ms TMA/15 s purge/30 ms water/15 s purge sequence. The deposition rate of Al_2O_3 was calibrated to be ~ 0.10 nm/cycle.

According to the literature, the LiPON film growth rate is relatively stable with the ALD temperature at 270–310 °C.[22] As the deposition temperature is increased, the concentration of carbon impurities in the LiPON film is decreased.[24] Therefore, the deposition temperature was maintained at 300 °C during the ALD process to achieve high film quality.[28]

Al-metal/LiPON–Al_2O_3 hybrid structure/ITO capacitor was fabricated for electrical measurements. Al metal top electrode was deposited by direct current magnetron sputtering by using a shadow mask with the diameter of 400 μm for each device. The ITO bottom electrode was grown on Si (100) by reactive sputtering (with an ITO target) before the ALD process. The Si wafer was degreased by isopropanol, with native oxide on the surface.

The XPS measurements were carried out on a PHI 5000 instrument with a monochromatic Al $K\alpha_1$ source of 1486.7 eV. The take-off angle was 45°. All high-resolution XPS spectra were collected with the pass energy of 23.50 eV, and the step size was 0.1 eV. All XPS spectra were referenced to the C 1s peak at 284.6 eV.[22] XPSPEAK 4.1 was used as the peak-fitting software for the deconvolution of the XPS spectra.[29] The high resolution TEM (HRTEM) images were taken by Talos F200X microscope operated at the acceleration voltage of 200 kV. Electrical measurements were performed using a Keysight E4990A impedance analyzer and Keithley 4200A-SCS semiconductor parameter analyzer in air.

3. Results and discussion

Figure 1 shows the high resolution XPS spectra (Li 1s, P 2p, N 1s, Al 2p, O 1s, and Si 2p) of ALD of the LiPON–Al_2O_3 hybrid structure. The Li 1s signal is seen below the detection limit of XPS for the hybrid structure. The sensitivity factor (ASF) of the Li 1s core level is 0.025, which is much lower than P 2p (ASF = 0.412) and N 1s (ASF = 0.477). It accounts for one of the reasons for the undetected Li 1s signal. The low concentration of Li atoms accounts for another reason for the lack of Li 1s peak. The relatively low concentration of Li 1s signal is also correlated to the organic ligands of precursors during the ALD process. These organic ligands may replace the –OLi group in the phosphazene chain during the ALD process.[27] The composition of the hybrid structure is quite different from that of the thick LiPON film with a thin Al_2O_3 overlayer (see supporting information, Fig. S1).[22] A trace amount of Si contamination is observed in this hybrid structure, which is from the undecomposed Li-precursor.

Figures 2(a) and 2(b) show large and small scale cross-section HRTEM images of the Al/LiPON–Al_2O_3 hybrid structure/ITO capacitor, respectively. No sharp delamination is observed in this HRTEM result. The LiPON–Al_2O_3 hybrid structure appears smooth interfaces with the electrode, with high roughness in cross-section view.

Figure 3(a) shows the schematic stack of the Al/LiPON–Al_2O_3 hybrid structure/ITO capacitor. Figure 3(b) shows the k vs. frequency (k–f) result. The k is 40 for the sample with the cycle ratio of 1:5 (LiPON vs. Al_2O_3) at 1 kHz, which is relatively higher than that of the Al_2O_3 film (~ 9). The k of the device is seen decreased with increased frequency, because the lithium ion polarization cannot catch up with the higher frequency applied. A platform with a dielectric constant of ~ 16 is seen in the frequency range of 20 kHz–100 kHz, which is higher than that of the Al_2O_3 matrix. It is hypothesized that the local displacement of ions from LiPON in samples contributes

additional polarization against the applied electrical field, resulting in larger k regards to the Al_2O_3 matrix. However, the dielectric constant is seen below 9 at 1 MHz. The degradation of k in the hybrid structure at high frequency is hypothesized to be from the incorporation of undecomposed organic ligands in the precursors (LiHMDs and DEPA). k can be calculated by the following equation:

$$k = (C \cdot d)/(\varepsilon_0 \cdot A),$$

where ε_0 is the permittivity of the substance, C represents the capacitance, d is the thickness of the film and A is the area of the top electrode.

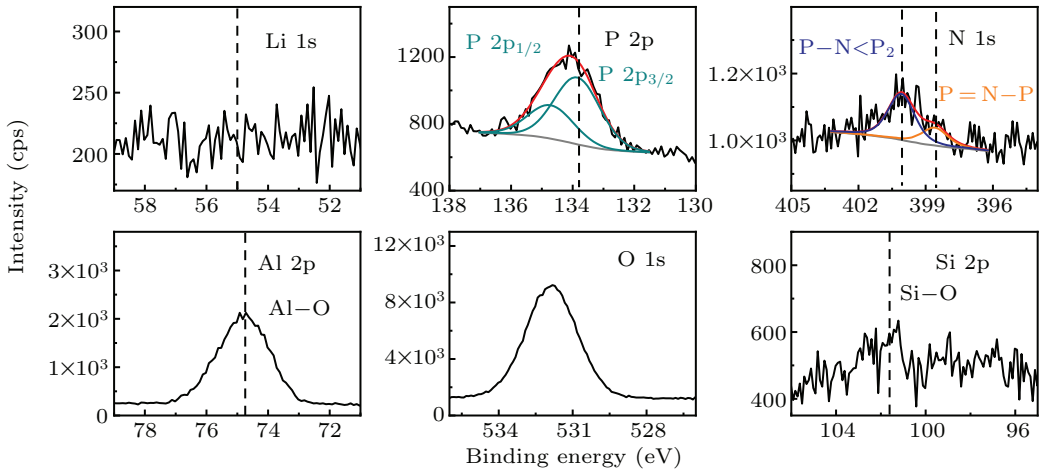


Fig. 1. High resolution core level spectra of Li 1s, P 2p, N 1s, Al 2p, O 1s, and Si 2p for LiPON–Al_2O_3 hybrid structure deposited at 300 °C.

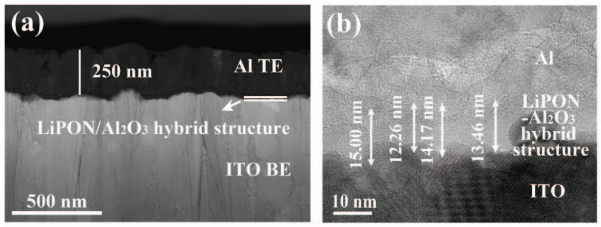


Fig. 2. (a) The large scale, (b) small scale cross-section HRTEM images for Al/LiPON–Al_2O_3 hybrid structure/ITO capacitor.

The degradation of k is also observed in other samples. The sample with the ALD cycle ratio of 1:1 is seen to possess the highest k among these samples in the frequency of 4 kHz–40 kHz. However, its k is below the sample with the ALD cycle ratio of 1:5 outside this frequency range. The k is the worst for the sample with the ALD cycle ratio of 1:10 (LiPON vs. Al_2O_3) for all frequencies, which may be from the low concentration of Li ions in the hybrid structure.

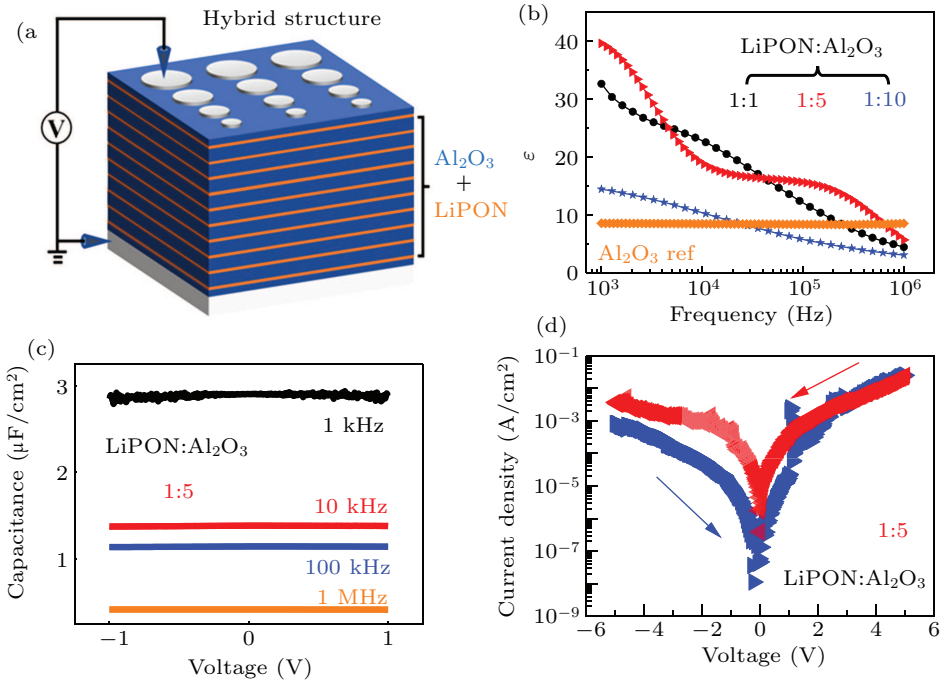


Fig. 3. (a) The schematic structure of Al/LiPON–Al_2O_3 hybrid structure/ITO capacitor and electric measurements, (b) k vs. frequency curve of these capacitors with ALD cycle ratio for LiPON:Al_2O_3 to be 1:1, 1:5 and 1:10, (c) capacitance versus voltage and (d) leakage current of the capacitor with the ALD cycle ratio for LiPON:Al_2O_3 to be 1:5.

The capacitance–voltage curve for the sample with the cycle ratio of 1:5 is shown in Fig. 3(c) at different frequencies. The current–voltage characteristic for the sample with the cycle ratio of 1:5 is shown in Fig. 3(d). Compared with the ion-incorporated Al_2O_3 reported in literature,[7] these electrolyte–dielectric hybrid structure materials also show excellent insulating properties (relatively lower leakage current). Although this hybrid structure is not suitable for traditional Si-based complementary metal-oxide-semiconductor architecture, it has the potential to serve as the dielectric in devices (such as thin-film transistors), which demands low-frequency application for future IoT systems.

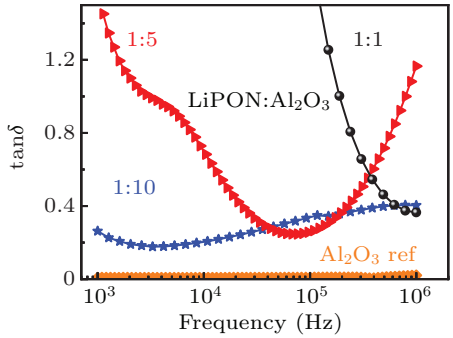


Fig. 4. The tan δ vs. frequency curves of these capacitors with ALD cycle ratio for LiPON:Al_2O_3 to be 1:1, 1:5, and 1:10, as well as Al_2O_3 matrix.

Figure 4 shows the loss tangent (tan δ) vs. frequency curves of these capacitors with ALD cycle ratio for LiPON:Al_2O_3 to be 1:1, 1:5, and 1:10, as well as Al_2O_3 matrix, respectively. Conductance loss and polarization loss are both contributed to the tan δ.[30–34] The movement of Li^+ under external voltage (polarization behavior) is responsible for the high tan δ in these hybrid structures than that in the Al_2O_3 matrix, which has been proved in literatures.[33,34] At low frequency ($\sim$ 1 kHz), tan δ of the hybrid structures increase as the concentration of LiPON in the hybrid structure increased. For samples (LiPON/Al_2O_3), tan δ is seen to decrease then increase with increasing frequency (except for the sample with the LiPON:Al_2O_3 ratio of 1:1, which is limited by the measurement frequency). Trend of tan δ vs. frequency for those hybrid structures is correlated with the concentration Li ions and the thickness of each LiPON layer (the movement of Li^+ is hypothesized to be limited by the Al_2O_3 layer). The tanδ of the LiPON–Al_2O_3 hybrid structure is large. However, consider tan δ of the hybrid structure is changed as the variation of the ALD cycle ratio of LiPON:Al_2O_3. Therefore, tan δ of this hybrid structure is expected to be further optimized by controlling the deposition condition, such as the ALD cycle ratio of LiPON and Al_2O_3, deposition temperature, precursor choice, pulse and purging time.

In order to determine which ion specie (Li or P/N) is responsible for the increased k in these samples, the dielectrics were grown only incorporated LiHMDs or DEPA instead of LiPON film, with the cycle number ratio of a certain precursor (LiHMDs/DEPA) to Al_2O_3 as 1:5. The recipe of the Al_2O_3 film is the same as the LiPON–Al_2O_3 hybrid structure. As shown in Fig. 5(a), LiHMDs only sample presents a strong frequency-dependent capacitance. The capacitance of the DEPA only sample maintains nearly constant through different frequencies. This indicates that the Li^+ contributes to the higher capacitance rather than the P or N related ions. Similar to the LiPON–Al_2O_3 hybrid structure, a platform characteristic of capacitance in the 5 kHz–30 kHz frequency range is also seen for the LiHMDs only sample. As shown in Fig. S4 (see supporting information), the capacitance of the sample with Li precursor only is 1.53 μF/cm^2 at 1 kHz, which is slightly lower than that of the sample with LiPON–Al_2O_3 (ALD cycle ratio of 1:5, shows capacitance of 2.90 μF/cm^2).

The leakage current density of LiHMDs only or DEPA only incorporated Al_2O_3 based capacitor are shown in Fig. 5(b), which is higher than that in the LiPON–Al_2O_3 hybrid structure (see supporting information, Fig. S5). Therefore, a degradation in terms of k and leakage current is seen for the sample with Li precursor only, compared to the sample with LiPON in the hybrid structure. This degradation can be explained that the LiHMDs and DEPA precursors react to form the LiPON layer during the ALD reaction process, resulting in less contaminants left in the hybrid structure film. However, a relatively higher concentration of remnant species is left in the LiHMDs only or DEPA only film, which is from the unreacted precursor (incomplete ALD reaction).

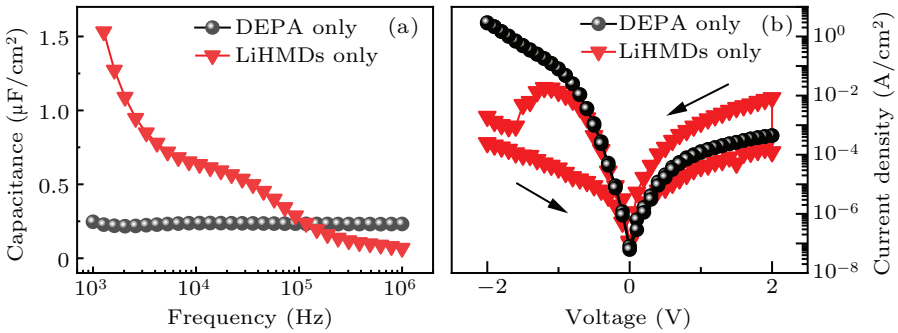


Fig. 5. (a) Frequency-dependent capacitance (C–f) curves and (b) leakage current curves for LiHMDs only or DEPA only samples.

In conventional dielectrics like Al_2O_3, HfO_2, k is mainly originated from the polarization of positive metal ions and negative oxygen ions shift in each crystal cell upon the electrical field. The remnant charges are located on the top and bottom surfaces for the film, associated with slight deformation of the bond length and direction.[16] In contrast, the polarization is originated from the displacement of Li^+ in the hybrid structure, but its movement is supposed to be limit by the Al_2O_3 layer (local shift upon electrical field). The Li ions are supposed to respond the electrical field easier, because it is not as strong bond with oxygen as the matrix metal ions (Al). The high sensitivity to the electrical field of the Li ions is likely the key factor to the fast response to the electrical field, which results in the high k property.

Compared to the EDL capacitor based on long-distance polarization,[2,13,35–37] this hybrid structure shows high k properties even at $\sim$ 100 kHz, indicating this hybrid structure device is close to the result in literature (ion in beta-Al_2O_3),[16] and suggests the ions in the hybrid films move locally upon the applied electrical field. It is consistent with the fact that the ALD of Al_2O_3 film is glassy and quite dense. Locally shifted ions can provide a significant contribution to k, as in the beta-Al_2O_3 films ($\sim$ 200).[16] The dielectric performance of the hybrid structure may be optimized with further effort.

4. Conclusion and perspectives

In conclusion, the $LiPONAl_2O_3$ hybrid structure is fabricated by *in situ* ALD process. The k of this hybrid structure is $\sim$ 40 at 1 kHz. A platform characteristic exists in this hybrid structure with the k of $\sim$ 16 in the frequency range of 20 kHz–100 kHz. The k of the hybrid structure is higher than that of the amorphous Al_2O_3 in the range of less than 100 kHz. The high k is likely to be originated from the locally shifted Li ions. This work sheds light on designing ultrahigh k dielectrics at less than 100 kHz range for low power devices in IoT applications via electrolyte/dielectric hybrid structure, compatible with semiconductor fabrication process.

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