Photonic curing of sol–gel derived HfO$_2$ dielectrics for organic field-effect transistors

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Abstract

An efficient way to reduce the supply voltages of organic field-effect transistors is the use of high-k inorganic materials. In order to allow high throughput during fabrication, solution-based processes for realizing inorganic dielectrics by using sol–gel procedures have become attractive in recent years. However, this procedure typically involves extended high-temperature annealing steps to achieve high-quality insulating layers which hampers fast fabrication and is incompatible to be carried out on low-temperature organic substrates. In this work, the use of a photonic curing technique is presented for the annealing of sol–gel derived hafnium oxide (HfO$_2$) dielectrics within a few seconds. The investigations demonstrate the reduction of the leakage current density of more than 3 orders of magnitude after the photonic curing process reaching only slightly higher values as obtained with dielectric films formed from highly sophisticated atomic layer deposition. Moreover, capacitance measurements reveal a dielectric constant of 26 indicating bulk-like properties. Furthermore, organic transistors based on photonic cured HfO$_2$ sol–gel dielectrics are fabricated and characterized operating at low voltages (< 2 V), low subthreshold swing (110 mV/decade) and charge carrier mobilities of 1 cm$^2$/Vs using a semiconducting liquid-crystal polymer.

Keywords: Organic field-effect transistors; Sol–gel; Photonic curing; Inorganic dielectrics; Solution-based processes

1. Introduction

A significant challenge in the realization of low-voltage and high-performance organic field-effect transistors (OFET) is the reduction of the cost factor especially when fabricating over large areas. In order to achieve low-voltage devices, the capacitance density of the dielectric has to be increased by first decreasing the dielectric thickness and/or using high-k materials. Both approaches are often limited when using organic dielectric materials since leakage currents drastically increase with decreasing thickness, and most organics do not exhibit high-k properties. This suggests the use of inorganic dielectrics may be more beneficial [1–4]. However, the deposition of high-quality inorganics for OFET devices is typically restricted to expensive high-vacuum processes such as sputtering [5–7], evaporation [8–10] or atomic layer deposition (ALD) [11–13] or other batch-to-batch processes, such as anodization [14–16], which hamper high throughput. One exception allowing the fabrication of inorganic films from solution which is compatible to technologies used in organic electronics, like roll-to-roll fabrication, is the sol–gel process [17]. In recent years, much work has been done in the investigation of sol–gel processed inorganic dielectrics for OFET. Examples include Al$_2$O$_3$ [18,19], ZrO$_2$ [20–22] or TiO$_2$ [23,24], but the most promising results have been achieved using HfO$_2$ [25–29]. Most of the sol–gel procedures involve extended temperature processing times beyond 400 °C to anneal the dielectric films to improve their electrical properties. This practice hinders fast throughput and makes it impossible to use low-temperature plastic substrates. However, the increased annealing temperature is essential for reducing the
leakage current in the dielectric which is an important quality factor for an insulating layer.

In this work we demonstrate the use of a photonic curing technology for the rapid annealing of HfO2 sol–gel layers to improve the dielectric properties within a few seconds. Furthermore, the investigations imply that this technology could provide the possibility to be carried out on cheap and low-temperature substrates, such as PET or PEN, due to the rapid thermal load limited to the surface of the substrate. In advance to this work, a quantitative comparison in terms of rapid thermal load limited to the surface of the substrate. In this work, we demonstrate the use of a photonic curing technology for the rapid annealing of HfO2 sol–gel layers to improve the dielectric properties within a few seconds. Furthermore, the investigations imply that this technology could provide the possibility to be carried out on cheap and low-temperature substrates, such as PET or PEN, due to the rapid thermal load limited to the surface of the substrate. In addition, the wafers were cleaned in an ultrasonic bath of titanium (10 nm) and platinum (100 nm) by e-beam evaporation. Afterwards, the wafers were cleaned in an ultrasonic bath in DI-water for 10 min respectively, rinsed in DI-water and dried. Afterwards, the wafers were cleaned in an ultrasonic bath in DI-water for 10 min respectively, rinsed in DI-water and dried under nitrogen. Subsequently, HfO2 sol–gel layers were deposited by spin-coating in two steps of 1000 rpm for 3 s and 3000 rpm for 30 s, baked on a hotplate at 150 °C for 5 min to remove solvent residues and treated by an additional thermal or photonic curing step. Then counter electrodes were deposited by thermal evaporation of gold through a shadowmask.

2.3. Organic transistor fabrication and characterization

Glass wafers with fully coated Ti/Pt gate electrode covered with the photonic cured HfO2 sol–gel derived dielectric were immersed in a tetradecylphosphonic acid (TDPA, Sigma-Aldrich) containing isopropanol solution (5 mmol/l) for 16 h in closed plastic beakers at 50 °C under constant stirring. Afterwards, the substrates were washed thoroughly in fresh isopropanol, blown dry with nitrogen and baked at 60 °C for 10 min on a hotplate to support chemisorption. The organic semiconductor FS111 from Flexink Ltd., U.K. was dissolved in chloroform at a concentration ratio of 10 mg/ml and filtered through a 0.2 μm filter. The solution was spin-coated at 2000 rpm for 20 s onto the SAM treated HfO2 dielectrics followed by a drying step of the films on a hotplate at 100 °C for 5 min. Subsequently, gold source/drain electrodes were formed by thermal evaporation through a shadowmask resulting in thicknesses of 100 nm. Characterization was carried out in ambient atmosphere and directly after fabrication using a Keithley 4200 semiconductor parameter analyzer. The field-effect mobility $\mu_{\text{sat}}$ was calculated in the saturation regime from the linear fit of $(I_D/J)^{1/2}$ versus $V_G$ using the equation

$$\mu_{\text{sat}} = \frac{L}{WC} \frac{\partial^2 I_{\text{Dsat}}}{\partial V_G^2}$$

where $L$, $W$, and $C_i$ are the channel length, channel width and capacitance per unit area of the dielectric respectively. The threshold voltage $V_T$ was extrapolated from the intercept point of the linear fit and the $V_G$-axis. An Agilent 4284A LCR meter was used for the capacitance determination at a frequency of 1 kHz. Subthreshold swing was calculated by taking the inverse of the slope $|I|_{\text{Dsat}}$ versus $V_G$ in the subthreshold regime.

2.4. AFM, ellipsometry, contact angle and FTIR characterization

AFM images were obtained in non-contact mode using a SIS ULTRAO objective with scanning speeds of 15 μm/s. Film thicknesses were measured using a Sentech SE 400adv ellipsometer with an average of eight points per wafer. Water contact angle values were taken using a Dataphysics OCA 20 optical contact angle measuring system and are an average of five measurements of three sets of samples. FTIR measurements were carried out using a Spectral Instruments Nicolet 6700 equipped with a grazing-angle reflectance accessory (SMART-SAGA). The spectra were recorded with a resolution of 4 cm$^{-1}$ and 1024 averaged scans.

XPS Characterization: XPS and UPS measurements were performed on a Kratos AXIS-165 multitechnique electron spectrometer system with a base pressure of $5 \times 10^{-10}$ Torr. Achromatic X-ray radiation of 1253.6 eV (Mg-K$_\alpha$) was used for the analysis.
utilized as the XPS excitation source for acquiring all XPS photoelectron spectra with a spot size of 400 μm x 300 μm.

2.5. SEM/FIB characterization

Cross-section images of the layer stacks were prepared using a Zeiss 1540 XB Cross Beam Scanning Electron Microscope.

3. Results and discussion

3.1. Choice of HfO₂ sol–gel procedure for photonic curing

A large number of synthesis procedures of HfO₂ sol–gels have been introduced in the last years by several research groups. In this work, four procedures showing the most promising approaches were evaluated to realize high quality inorganic dielectrics. The procedures mainly differ in the utilized precursors which are hafnium tetrachloride [30], hafnium ethoxide [31], hafnium pentanedionate [32] and hafnium isopropoxide [33]. The synthesis of the sol–gels was carried out as described in the respective publications and typically involved dissolving the precursor in a solvent followed by a reflux reaction at elevated temperatures to promote hydrolysis and polycondensation reactions. Spin-coating was chosen as the optimal deposition technique to achieve uniform thin-films of the sol–gel layers. The deposition was carried out on 4-inch borosilicate glass wafers which were fully coated with 10 nm of titanium and 100 nm of platinum acting as the bottom electrode. The reason to use a noble metal as the base electrode was to avoid the formation of additional interfacial layers which can drastically influence the insulating properties of the metal–insulator–metal stack and thus distort the leakage current and capacitance measurements. The native oxides of silicon or aluminium for instance have been shown to exhibit significant insulating behavior despite being only a few nanometers thick [34]. Due to the fact that the sol–gels differ in concentration, the thicknesses of the deposited sol–gel films varied significantly for each procedure. A single spin-coating layer of hafnium tetrachloride, hafnium pentanedionate, hafnium ethoxide and hafnium isopropoxide resulted in layer thicknesses of 55 nm, 30 nm, 32 nm and 12 nm respectively as measured by ellipsometry. Multiple spin-coatings were used to adjust the thickness for all sol–gel layers to approximately 60 nm thereby allowing a quantitative comparison. Afterwards, the sol–gel layers were annealed at 200 °C in combination with UV/Ozone for 1 h. This procedure has been shown to be an effective way to improve the densification and hence the quality of the inorganic HfO₂ layers at lower temperatures [35,36]. The main evaluation criterion for the quality of the sol–gel layers is the insulation behavior, and it can be characterized by measuring the leakage current density through the HfO₂ films. To evaluate this, capacitor structures were fabricated by thermally evaporating gold top-electrodes with areas of 1 mm² through a shadow mask on the substrates. Leakage current measurements for each of these sol–gel procedures are presented in Fig. 1. The diagram clearly shows that the sol–gel layer based on the precursor hafnium isopropoxide has the lowest leakage current by several orders of magnitude, e.g. 10⁻⁵ A/cm² at a bias voltage of 5 V, followed by hafnium chloride, hafnium ethoxide and hafnium pentanedionate. In all cases, this value is still too high to use such films as the dielectric in OFETs. For comparison, the leakage current densities for HfO₂ films deposited by ALD in similar thicknesses are typically in the range of 10⁻⁹ A/cm² [37]. This indicates that the sol–gel films are not completely converted into pure HfO₂ since the annealing temperature was kept relatively low. Nevertheless, the sol–gel process involving hafnium isopropoxide seems to be a promising starting procedure for further investigations using photonic curing.

3.2. Photonic curing of sol–gel derived dielectric thin-films

Parallel experiments with hafnium isopropoxide sol–gel layers annealed at 600 °C in a convection oven verified a further decrease of the leakage currents down to 10⁻⁹ A/cm² (see Fig. S1). Thus, even higher temperatures would have led to an optimal curing of the HfO₂ layer with leakage currents in the range of the targeted values of high quality films. However, conventional equilibrium annealing processes, such as hot-plates, convection ovens, etc., at such high temperatures exceed the maximum working temperature of any known organic, flexible substrate. Moreover, these processes are also time-consuming. Instead, a new approach, called photonic curing, was followed [38]. This new process uses a high intensity flashlamp that is digitally controlled to modulate and tune the power delivery to a particular film/substrate combination. It allows a rapid, high-temperature (above 1000 °C) treatment of functional thin-films on low-temperature substrates. Due to the fact that organic substrates like PET, PEN, and even glass do not readily absorb light, the temperature here is held relatively low compared to the thin-films above which can prevent damage. However, the temperature in the interface between the thin-film and substrate can reach values far beyond the maximum acceptable temperature for the substrate.
However, since the time exposure is in the range of milliseconds there is not enough time for its properties to be significantly changed. Furthermore, thermal conduction into the bulk of the substrate supports a rapid cooling of the thin-film after exposure due to conduction of the applied thermal energy into the bulk. In that way, the thermal equilibrium is reached within several milliseconds preventing any damage of the substrate material.

For the experiments using the photonic curing technology, glass wafers were coated with titanium and platinum by electron beam evaporation acting as the bottom electrode. Subsequently, the sol–gel solution based on hafnium isopropoxide was spin-coated on the glass wafers and dried at 150 °C for 5 min to remove solvent residue. The spin-coating process was repeated six times to reach a final thickness of the HfO2 layer of 70 nm. Atomic Force Microscopy (AFM) revealed a smooth surface of the sol–gel layer with a root-mean square (rms) roughness of 0.3 nm (see Fig. S2(a)).

All photonic curing work was performed on a PulseForge 3300 photonic curing system manufactured by NovaCentrix. This flashlamp based system has the capability of arbitrarily changing the intensity, pulse length, and even the temporal evolution of the pulse shape. Pilot tests of the HfO2 photonic curing revealed that the pulse condition yielding the best results was achieved by using an engineered pulse profile (shaped pulse) rather than a single pulse. In short, a single pulse of light with sufficient power and radiant exposure to affect the film failed the substrate by inducing surface cracks, or in some cases, shattered the glass substrate. For the condition which yielded the best results, an exposure energy of 10.0 J/cm² at a peak power of 15 kW/cm² was irradiated on the film. The shaped pulse used was a three pulse modulation with the timing and temporal evolution shown in Fig. 2. It can be seen that the exposure time of the shaped pulse was set to less than 2 ms. The glass wafers with the HfO2 sol–gel film were placed on a conveyor at 6 feet per minute. Three shaped pulses were exposed to the sample at approximately 1.2 Hz while it was being conveyed to achieve a uniform cure. A white light source with a spectrum similar to the PulseForge beam was used to measure the reflectance of the surface of the wafer to be approximately 40%. Thus, 6 J/cm² was absorbed in the sol–gel film, not the 10 J/cm² measured from the beam. The emitted power in the beam was 15 kW/cm², so the peak radiated power absorbed in the film was 9 kW/cm².

The temperature profile in time within the thin film stack was modeled using SimPulse, manufactured by NovaCentrix. This is a machine integrated thin film stack thermal modeling program that accounts for the machine settings, the electrical performance, and optical efficiencies of the flashlamp system and combines with that an interactive thin film stack thermal modeler to ultimately output the thermal response of the thin film stack to that particular pulse sequence emitted by the tool. Typically, SimPulse is run in volumetric mode in which the Lambert–Beer law is used to model the absorption of the light as a function of depth in order to calculate the temperatures within the layers during and after the pulse sequence [39].

Fig. 3 presents the temperature distribution of the layer stack as a result of the simulation. The graphs show the temperature evolved on the surface of the sol–gel layer, 50 µm beneath the sol–gel layer in the glass substrate and on the backside of the glass substrate (50 µm from top surface). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)
substrate. It is interesting to note that a peak temperature of nearly 1200 °C was achieved on the sol–gel layer surface and less than 400 °C in the glass substrate after the shaped pulses. Moreover, the simulation suggests that the whole stack cooled down to a temperature below 200 °C within only 20 ms after each shaped pulse. The final equilibrium temperature of the wafer reached approximately 140 °C after less than 2 s.

In the experiments, a clear color change of the HfO2 was visible after the curing and changed from blue to light brown as shown in Fig. 4. This is attributed to a densification of the layer. In order to verify this assumption, cross-section pictures of the stack before and after photonic curing were taken by the use of FIB/REM as presented in Fig. 5. The images show that the sol–gel layer had a thickness of 71 nm prior to the photonic curing which decreased to a thickness of 62 nm afterwards confirming a densification step. AFM measurements showed smooth surfaces of the sol–gel layers after photonic curing with an rms roughness of 0.4 nm (see Fig. S2(b)). Interestingly, the surface roughness was found to be slightly lower on sol–gel layers using the photonic curing than on sol–gel layers using a thermal curing process at 600 °C for 1 h in a convection oven which showed an rms roughness of 0.9 nm.

In addition to the optical measurements, electron spectroscopy for chemical analysis (ESCA) was used to examine the atomic composition of the layers. Fig. 6 presents the spectra of the HfO2 layer before and after photonic curing. It can be seen that the atomic percent of carbon decreased after the photonic curing of about 5% which means that the organo-metallic precursor is further converted into HfO2. Additionally, the composition of the cured HfO2 is close to stoichiometric with 28 at% of Hf and 55 at% of O. The still high amount of carbon in the cured layers could either arise from organic residues of the sol–gel solution or adsorbates on the surface of the samples since the detection of specific atoms in a layer is limited to only several nanometers due to scattering of emitted electrons. The penetration depth of the X-rays, or more precisely, the detection limit of emitted electrons is around 5 nm with the ESCA measurement system used in this investigation.

In order to characterize the electrical properties of the photonic cured sol–gel layers, capacitor structures were fabricated by evaporating gold through a shadow mask to form top-electrodes with an area of 1 mm². The results of leakage current measurements through the HfO2 sol–gel layers before and after the photonic curing are presented in Fig. 7a. The diagram shows that the leakage current density is decreased about more than 3 orders of magnitude after the photonic curing process down to $10^{-7}$ A/cm². This is nearly 2 orders lower than a sol–gel film annealed at 200 °C in combination with UV/ozone. This result is encouraging since the values for the leakage current are close to data reported in literature for ALD deposited HfO2 exhibiting leakage current densities of $10^{-9}$ A/cm² which was fabricated in a more sophisticated process with large efforts [13]. In addition, the capacitance measurements of the photonic cured sol–gel layers presented in Fig. 7b as well revealed a dielectric constant of 26 which is bulk-like for this material [40].

### 3.3. Organic transistors based on sol–gel derived HfO2 dielectrics

In order to evaluate the feasibility of using sol–gel derived HfO2 dielectrics treated by a photonic-curing process in an OFET, bottom-gate top-contact devices were fabricated and characterized based on the organic semiconductor FS111 provided by the company Flexink. This material is a proprietary donor–acceptor co-polymer exhibiting a HOMO level of 5.2 eV and has a number average molecular mass ($M_n$) of 39,700 g/mol, and a polydispersity of 2.53. Promising results

![Photonically cured area](image1)

![Untreated area](image2)

Fig. 4. Partially annealed HfO2 sol–gel coated glass wafer using a photonic curing process.

![Cross-section](image3)

Fig. 5. Cross-section of a HfO2 dielectric layer formed by a sol–gel process before (left) and after the photonic curing (right) verifying a densification step.
have been already achieved with preceding versions of this liquid-crystal polymer showing comparable performances to 6,13-bis(triisopropylsilylethynyl)pentacene with high device-to-device reproducibility [41]. Prior to semiconductor deposition the HfO$_2$ dielectrics were treated with tetradecylphosphonic acid (TDPA) since the use of this Self-Assembled Monolayer (SAM) on inorganic dielectrics has shown to improve the OFET performance remarkably [42]. Several reports indicate that SAMs decrease surface roughness and diminish the density of trapping sites at the semiconductor/dielectric interface which directly enhances charge-carrier mobility [43–45]. The presence of the SAM was verified by Fourier Transform Infrared Spectroscopy (FTIR) and water contact angle measurements. In order to evaluate the quality of a SAM consisting of molecules with a methylene chain backbone, it has been shown that the peak frequencies in an FTIR spectrum for the symmetric and asymmetric methylene stretches, $\nu_s$(CH$_2$) and $\nu_a$(CH$_2$) respectively, are good indicators to distinguish between ordered (crystalline-like) or disordered (liquid-like) structures [46,47]. A crystalline-like structure is characterized by having alkyl chains in an all-trans conformation with characteristic peaks at $\nu_a$(CH$_2$) $\leq$ 2918 cm$^{-1}$ and $\nu_a$(CH$_2$) $\leq$ 2850 cm$^{-1}$ whereas a liquid-like layer is characterized with the two absorption bands shifting to higher frequencies. As presented in Fig. 8 the peak frequencies of $\nu_s$(CH$_2$) and $\nu_a$(CH$_2$) appear at 2850 cm$^{-1}$ and 2917 cm$^{-1}$ respectively indicating the formation of well-ordered TDPA SAM on photonically cured HfO$_2$ sol–gel layers. In addition, the water contact angle increased from 7$^\circ$ for the untreated dielectric to 110$^\circ$ for the SAM-treated dielectric (see Fig. S3). The rms roughness of the TDPA treated sol–gel layers slightly increased to a value of 0.6 nm as observed in AFM measurements which is negligible and still represents a smooth surface (see Fig. S2(d)).

Spin-coating of the organic semiconductor on such highly hydrophobic surfaces was accomplished by using chloroform as the solvent. Due to the low boiling point, low surface tension and high solubility of chloroform the formation of homogenous semiconductor layers with good coverage on the SAM-treated HfO$_2$ was possible. The transistor fabrication was finalized by evaporating gold source/drain electrodes through a shadow mask onto the organic semiconductor. The devices

![Graph](image_url)

Fig. 6. ESCA spectra showing the chemical composition of HfO$_2$ dielectric layer formed by a sol–gel process before (a) and after the photonic curing (b) showing a reduction of the carbon content after curing suggesting a further elimination of organic components in the sol–gel film.

![Graph](image_url)

Fig. 7. Comparison of the leakage current densities of sol–gel derived HfO$_2$ dielectrics before and after the photonic curing (a) and capacitance measurement of a photonically cured dielectric structure (b).
were subsequently characterized by measuring the transfer and output curve in the dark using a semiconductor parameter analyzer. Representative transfer [i.e., drain current ($I_D$) versus gate voltage ($V_G$)] at a fixed drain voltage ($V_{DS}$) of $-2$ V and output curves [i.e., $I_D$ versus $V_D$ measured at different $V_G$] for a transistor having a channel length of $100 \mu$m and width of $3.5$ mm is presented in Fig. 9. As can be seen in the transfer curve, the transistor shows a reliable switching behavior with on/off current ratios of $4 \times 10^5$ and negligible hysteresis. Due to the high-k property of the dielectric, the supply voltages were in the range of $-2$ V. In addition, parameter extraction of the mobility and threshold voltage yielded in values of $1 \text{cm}^2/\text{Vs}$ and $-0.8$ V respectively discovering high charge carrier mobility for FS111. Furthermore, low subthreshold swing of $110 \text{mV/decade}$ was extracted which is close to the theoretical room-temperature limit of $57 \text{mV/decade}$ [48]. All these findings emphasize the good interaction of sol–gel processed gate-dielectrics in combination with Self-Assembled Monolayers using a solution-deposited organic semiconductor. However, one drawback becomes obvious in the output curve in which the characteristics do not cross the point of origin correctly indicating increased leakage currents through the dielectric. At $V_G$ of $-2$ V and $V_{DS}$ of $0$ V, the drain current was measured with approximately $1 \mu$A which indicates insufficient insulation of the hybrid dielectric. We believe that the unpatterned gate and organic semiconductor lead to an increased leakage between gate and drain due to additional leakage paths through the dielectric resulting in the distortion of the output curve in the linear regime. Patterning of both, the gate and the organic semiconductor, has been shown to be an effective way to reduce this effect [49]. Nevertheless, the curves reach satisfying saturation and the overall functionality of the transistor is given as seen in both diagrams.

4. Conclusion

In this work, novel approaches were examined to realize high quality inorganic films from solution using a rapid photonic curing process. In order to set an optimal starting position, the quality of conventionally cured HfO$_2$ layers derived from several sol–gel procedures were compared and evaluated in terms of their electrical properties. The most promising procedure including the precursor hafnium isopropoxide was chosen for the photonic treatment and the chemical compositions as well as the electrical properties of the layers before and after photonic curing were analyzed. In parallel to the experiments, a thermal simulation of the photonic treatment was carried out revealing temperature peaks up to $1200 \text{C}$ in the sol–gel layers. However, the thermal load of the substrate was held low due to a rapid cooling below $200 \text{C}$ in less than $20 \text{ms}$ after each photonic curing step. The simulations suggest a thermal equilibrium of the layer stack at approximately $140 \text{C}$ after $2 \text{s}$ when using $3$ sequenced shaped pulses which is encouraging for using this technology on low-temperature, organic substrates in future experiments. The experimental results verified a significant improvement of the HfO$_2$ layers after the photonic curing showing bulk-like properties and smooth surfaces. In addition, these layers were treated with a Self-Assembled Monolayer and integrated into an organic transistor. Charge carrier mobilities of $1 \text{cm}^2/\text{Vs}$ at low supply voltages were achieved using the semiconductor FS111. Increased leakage currents are observed but these effects can
be attributed to non-optimized transistor layout due to unoptimized gate and organic semiconductor layers.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.ceramint.2014.07.099.

References


