Post Deposition Annealing Atmosphere Effect on Performance of Solid State Incandescent Light Emitting Device

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Electrical and optical properties of solid state incandescent light emitting devices made from MOS capacitors of the same Zr-doped HfO2 high-k film but different post deposition annealing atmospheres have been investigated. Compared with the N2 annealed sample, the O2 annealed capacitor has a thicker and more SiO2-rich interface layer, lower defect densities, a larger dielectric breakdown strength, and a lower leakage current. Light emitted from the solid-state incandescent light emitting device made from the O2 annealed capacitor has a high intensity and slightly more blue component than that made from the N2 annealed capacitor due to the higher temperature of the conductive path. The former contains more Si and O elements in conductive paths than the latter does, which can be used to explain the difference in their electrical and optical characteristics. The post deposition atmosphere affected the original high-k stack’s material and physical properties, which influences the formation and material composition of conductive paths and eventually the light emission characteristics.

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If incandescent and fluorescent light lamps are replaced with solid-state light emitting devices (LEDs), the energy consumption from lighting in the United States can be saved by about 38%.1 LEDs are usually made of direct-bandgap semiconductor materials prepared in the p-n junction or quantum well (QW) structure.2,3 They require the growth of high quality compound semiconductor epitaxy layers on single crystal substrates. The process is expensive and tedious.1–3 The light emission principle is the electron-hole or exciton-exciton recombination. The energy of the emitted light is equivalent to that of the bandgap energy of the semiconductor material.1,4 Therefore, the broad-band light cannot be emitted from a single LED. The white light can be generated using 3 different kinds of LEDs, i.e., red, green, and blue light emitting, or a blue LED in combination with a phosphor layer.5 CdSe/ZnS core-shell nanocrystals have been used to convert the blue/UV light into the green/red light, which is also a method of generating the white light.6 However, the size of the nanocrystal has to be tightly controlled to emit a specific color, which is difficult to achieve.7 The semiconductor based LED is often subject to the “droop” failure mechanism that shortens the lifetime.8 It is desirable to have a LED that emits the broad band white light from a single device and can be fabricated with the IC-compatible process with a long lifetime. Recently, Kuo’s group reported a new type of solid-state incandescent LED (SSI-LED) that emitted the warm white light similar to that of the incandescent bulbs.9–16 The light emitting principle of the SSI-LED is the thermal excitation of the nanosized conductive path from the passage of a current.9,10 It is made from a MOS capacitor composed of an amorphous high-k dielectric thin film, such as Zr-doped HfO2 (ZrHfO), HfO2, or WO3, on a p-type Si wafer.9,16 These high-k dielectrics are often used in the MOS field-effect transistors (MOSFETs) or capacitors. Upon the deposition of the high-k dielectric film, a post deposition annealing (PDA) step is carried out to densify and to reduce defects in the film. The PDA condition is critical to the material and electrical properties of the final device.17,18 For the SSI-LED, the PDA process influences the conductive path formation and therefore, characteristics of the emitted light.11,13,19 In this paper, authors investigated the effect of the PDA atmosphere on the electrical and optical properties of the SSI-LED.

MOS capacitors composed of the same ZrHfO high-k dielectric thin film but different PDA atmospheres were fabricated on dilute HF cleaned p-type (1015 cm−3) <100> Si wafers. The ZrHfO film was sputter-deposited from a Zr/Hf (12/88 wt%) target at Ar/O2 (1:1) atmosphere at 5 mTorr and 60 W for 12 min. Subsequently, the sample was processed with a PDA step at 800 °C for 3 min under the N2 or O2 atmosphere using a rapid thermal annealing (RTA) equipment. A Hf-silicate (HfSiOx) interface layer was formed between the amorphous bulk ZrHfO layer and the Si substrate, which was confirmed from the electron spectroscopy for chemical analysis (ESCA) and the high resolution transmission electron spectroscopy (HRTEM).18,20 Then, an 80 nm thick ITO film was deposited on top of the ZrHfO film and wet-etched into 300 μm diameter gate electrodes. An aluminum film was deposited on the back side of the wafer to form the ohmic contact. The complete sample was annealed at 400 °C under H2/N2 (1:9) atmosphere for 5 min. The sample’s capacitance-voltage (C-V) and leakage current-voltage (I-V) curves were measured using an Agilent 4284A LCR meter and an Agilent 4140B PA meter/DC voltage source, respectively. The equivalent oxide thickness (EOT), flatband voltage (VFB), forward V-I and leakage I-V, and oxide trapping density (Qot) were extracted from the C-V curve using the NCSU CVC program.21 To form the SSI-LED, the capacitor was first applied with a gate voltage (Vg) larger than the breakdown voltage (Vbd) to form conductive paths. The detailed description of the conductive path formation and characterization can be found in Refs. 9-15. Subsequently, when a Vg was applied to the ITO gate electrode, the conductive paths were excited to emit light. The emission spectrum was measured with an optical emission spectrometer (StellarNet BLK-C-SR-TEC) through an optical fiber located about 2 mm above the ITO electrode. The 1931 CIE coordinates of the emitted light were calculated using the color matching function.22 They were transferred to the CIE 1960 chart to calculate the color correlated temperature (CCT).23 The color rendering index (CRI) was calculated using the NIST CQS 7.4 program.24 The elemental profile of the sample was measured from the top of the ITO electrode to the Si substrate with the secondary ion mass spectrometry (SIMS, CAMECA IMS 4f ion microprobe) using the Cs+ ion beam at 5.5 keV and the incident angle of 43° in the positive charge secondary ion mode. The signal was obtained from an area of 50 μm × 50 μm on the specimen.

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Results and Discussion

Figure 1 shows C-V hysteresis curves of N2 and O2 PDA capacitors with $V_g$ swept from $-2$ V to $+1$ V (forward) and then back to $-2$ V (backward) at 1 MHz. Table I lists electrical parameters calculated from these C-V curves. Both samples show negligibly small hysteresis windows, i.e., $\Delta V_{FB} = 0.0031$ V and 0.0025 V for the N2 and O2 PDA conditions, respectively. In the accumulation region, the N2 PDA sample has a larger capacitance than the O2 PDA sample does, i.e., $2.01 \times 10^{-10}$ F vs. $1.87 \times 10^{-10}$ F. Since the EOT of the former is smaller than that of the latter, i.e., 11.12 nm vs. 12.03 nm, there are two possible explanations for this result. First, since the HfSiOx interface layer of the O2 PDA sample is more SiOx-rich than that of the N2 PDA sample, the former has a lower effective $k$ value than the latter. Second, the larger total physical thickness of the O2 PDA sample contributes to its lower capacitance. Furthermore, the $D_h$ and $Q_{ot}$ of the O2 PDA sample, i.e., $1.61 \times 10^{11}$ cm$^{-2}$ - eV$^{-1}$ and $5.21 \times 10^{11}$ cm$^{-2}$, separately, are smaller than those of the N2 PDA sample, i.e., $1.80 \times 10^{11}$ cm$^{-2}$ - eV$^{-1}$ and $7.13 \times 10^{10}$ cm$^{-2}$, separately. The same trend was observed in other similar devices. Therefore, the PDA atmosphere is an important factor in determining the dielectric properties of the capacitor that will be broken down to form the SSI-LED.

Figure 2 shows I-V curves of N2 and O2 PDA capacitors stressed from 0 V to $-10$ V. Each curve shows the apparent breakdown phenomenon, i.e., the current jumps abruptly by several orders of magnitude at $V_{BD}$. The magnitude of the $V_{BD}$ of the N2 PDA sample is smaller than that of the O2 PDA sample, i.e., $-5.65$ V vs. $-6.1$ V, which may be contributed by the difference in their physical thicknesses. Also, the breakdown strength of the ZrHfO stack is related to the defect density in the original film. Since the O2 PDA sample has fewer defects in the ZrHfO bulk and HfSiOx interface layers than the N2 PDA sample has, the former is more difficult to break down to form conductive paths than the latter. The breakdown is probably due to the occurrence of the SSI-LED. However, Fig. 4c shows that the leakage current of the O2 PDA sample is lower than that of the N2 PDA sample. Therefore, conductive paths in the O2 PDA sample are more effectively excited than those in the N2 PDA sample.

It is difficult to measure the light emission efficiency of a single SSI-LED in Fig. 4 because of the small size, i.e., 300 $\mu$m in diameter, and the lack of a proper characterization equipment. Assuming that light is emitted isotopically from the ITO electrode in the hemisphere shape, the relative light emitting efficiencies of the two samples can be compared from their emission spectra because they are measured under the same condition. The external quantum efficiency (EQE) of a SSI-LED can be estimated by dividing the number of emitted photons (NP) with the total number of injected electrons using the following equation:

$$\text{EQE} = \frac{\text{NP}}{\text{NP}_{inj}}$$

Table I. Parameters calculated from C-V hysteresis curves of the N2 and O2 PDA samples.

| PDA condition | C (F) | EOT (nm) | $\Delta V_{FB}$ (V) | $D_h$ (cm$^{-2}$ - eV$^{-1}$) | $Q_{ot}$ (cm$^{-2}$) |
|---------------|------|---------|---------------------|-----------------|-----------------|
| N2 PDA        | $2.01 \times 10^{-10}$ | 11.12 | 0.0031               | $1.80 \times 10^{11}$ | 7.13 $\times 10^{9}$ |
| O2 PDA        | $1.87 \times 10^{-10}$ | 12.03 | 0.0025               | $1.61 \times 10^{11}$ | 5.21 $\times 10^{9}$ |

Figure 1. C-V hysteresis curves of N2 and O2 PDA capacitors. $V_g$ swept from $-2$ V to $+1$ V to $-2$ V at 1 MHz.

Figure 2. I-V curves of the N2 and O2 PDA capacitors stressed with $V_g$ swept from 0 V to $-10$ V.
Figure 3. High-magnification photos of light emissions from (a) N₂ and (b) O₂ PDA SSI-LEDs at V_g = -20 V, -30 V, -40 V, and -50 V.

Following equations

\[
EQE = \frac{NP}{I/q} \quad [1]
\]

\[
NP = \frac{2\pi r^2}{A_{slit}} \int E(\lambda) d\lambda \quad [2]
\]

where \( I \) is the current in amps (A), \( q \) is the charge of an electron, \( 2\pi r^2/A_{slit} \) is the factor of fraction of light collected by an entrance slit, \( E(\lambda) \) is the irradiance of the light source, \( \lambda \) is the light wavelength, \( h \) is the Planck constant, and \( c \) is the speed of light. The detailed measurement and calculation of EQE’s can be found in Ref. 29. Figure 5 shows relative EQE’s of SSI-LEDs (in arbitrary unit) of the O₂ and N₂ PDA SSI-LEDs. The actual EQE’s of these devices should be much larger than those in the figure because the spectra were collected...
through the tip of an optical fiber about 2 mm above the surface of the ITO electrode. At the same $V_g$, the EQE of the O$_2$ PDA sample is consistently higher than that of the N$_2$ PDA sample. Since the light emission efficiency of a SSI-LED is a complicated function of the composition, size, and distribution of conductive paths, more studies are required to identify the actual factors contributing to the EQE difference.

Figure 6 shows emission spectra of the SSI-LEDs in Fig. 4 normalized with the peak intensities. They almost overlap except the very slight blueshift of the O$_2$ PDA sample and the very small redshift of the N$_2$ PDA sample. This phenomenon is consistent with the previous observation that dots in Fig. 4b appeared to be brighter and whiter than those in Fig. 4a. It also indicates that the same light emission principle, i.e., thermal excitation of the conductive path, is applicable to both N$_2$ and O$_2$ PDA samples.

The CCT’s and CRI’s of emission spectra of Fig. 5 samples stressed at different $V_g$’s have been calculated and summarized in Table II. Both samples have high CRI $R_\text{a}$’s, i.e., 94.4–98.6 for the N$_2$ PDA SSI-LED and 93.9–98.3 for the O$_2$ PDA SSI-LED. They are close to those of the incandescent light bulb that emits light based on black body emission. The $R_\text{a}$ values also increase with the increase of $|V_g|$. The negative shift of the $C-V$ curve is due to the generation of hole-trapping defects in the breakdown process. The original O$_2$ PDA capacitor has better electrical properties than those of the original N$_2$ SSI-LED approach those of the black body emission with the increase of the applied voltage. At the same time, the CCT increases with the increase of $|V_g|$, which is also consistent with the phenomena of thermal excitation generated light emission. Compared with the N$_2$ PDA SSI-LED at the same $V_g$, the O$_2$ PDA SSI-LED has a slightly higher CCT and the color coordinate is closer to the blue light region. Therefore, conductive paths in the O$_2$ PDA SSI-LED have a higher local temperature and emit the light with higher energy than those in the N$_2$ PDA SSI-LED do. The smaller CRI $R_\text{a}$ values of the O$_2$ PDA SSI-LED also reflect the rendering of light in the red color region.

To further study the function of the conductive path in the ZrHfO high-k stack, samples at three different process steps were characterized: (a) fresh MOS capacitors before breakdown, (b) devices immediately after breakdown, i.e., with a quick $V_g$ sweep from 0 V to $-10$ V, and (c) devices after being stressed at $V_g = -20$ V for 20 min. Figure 8 shows $C-V$ curves of (a) N$_2$ and (b) O$_2$ PDA samples, separately, measured by sweeping $V_g$ from $-2$ V to $+1$ V at 1 MHz. After the initial breakdown, the shape of the $C-V$ curve is similar to that of the fresh capacitor except the negative shift and the lower capacitance. Immediately after the breakdown, the conductive path is not fully developed, i.e., the resistance is still high in the low $V_g$ range. Therefore, the complete device behaves like a low-quality capacitor.

The negative shift of the $C-V$ curve is due to the generation of hole-trapping defects in the breakdown process. The original O$_2$ PDA capacitor has better electrical properties than those of the original N$_2$
PDA capacitor. For example, in addition to a larger $V_{BD}$ as shown in Fig. 2, the former has a smaller $D_p$ than the latter, i.e., $1.61 \times 10^{11}$ cm$^{-2}$·eV$^{-1}$ vs. $1.80 \times 10^{11}$ cm$^{-2}$·eV$^{-1}$. Immediately after the breakdown, the O$_2$ PDA sample remains a better capacitor than the N$_2$ PDA sample does, e.g., a smaller C-V shift of 0.5 V instead of 0.85 V. This result is consistent with the smaller leakage current of the former compared with that of the latter, as shown in Fig. 2. After the sample is stressed with a voltage much larger than the $V_{BD}$ for a long period, i.e., at $-20$ V for 20 min, conductive paths are fully developed and the high-$k$ stack loses the insulating capability. The C-V curve of the N$_2$ PDA sample is flatter than that of the O$_2$ PDA sample because of the formation of more conductive paths in the former.

Since electrical properties of the MOS capacitor vary with the PDA atmosphere, the composition of the high-$k$ stack should change accordingly. Figure 9 shows SIMS profiles of In, O, Zr, Si, and SiO elements in (a) N$_2$ and (b) O$_2$ PDA capacitors before the breakdown. For both samples, the $^{115}$In$^+$ signal drops sharply at the ITO/ZrHfO interface. Therefore, the PDA atmosphere has little effect on the reaction between ITO and ZrHfO. On the other hand, the $^{16}$O$^+$ signal drops gradually across the ZrHfO layer until the Si substrate. Currently, it is difficult to identify the source of $^{16}$O$^+$, which can come from ITO, ZrHfO, or the O$_2$ PDA gas. The $^{90}$Zr$^+$ signal is only detected in the ZrHfO high-$k$ stack. The $^{28}$Si$^{16}$O$^+$ and $^{28}$Si$^{18}$O$^+$ signals are located at the ZrHfO/Si interface because their peaks are closer to the Si substrate than the $^{90}$Zr$^+$ peak is. Therefore, the $^{28}$Si$^{16}$O$^+$ component must be from the HiSiO$_x$ interface layer formed between the bulk ZrHfO and the Si substrate. The above elements are good references for the comparison of the O$_2$ and N$_2$ PDA samples. The peak ratio of $^{28}$Si$^{16}$O$^+$ in the interface layer to $^{28}$Si$^+$ in the Si substrate of the O$_2$ PDA sample is larger than that of the N$_2$ PDA sample, i.e., 2.21 vs. 1.59. There-fore, the HiSiO$_x$ interface layer in the former is more SiO$_x$-rich than that in the latter, which is consistent with the ESCA result on similar samples.

Moreover, the $^{28}$Si$^{16}$O$^+$ signal in the O$_2$ PDA sample is slightly broader than that in the N$_2$ PDA sample, which can be explained by the former’s thicker interface layer. During the annealing, O$_2$ could diffuse through the high-$k$ film to reach and react with the Si substrate to facilitate the growth of the interface layer.

Previously, it was proved that conductive paths were physically formed in the high-$k$ stack after the dielectric breakdown process. Therefore, compositions of the high-$k$ stack before and after the formation of conductive paths can be different. Figure 10 shows SIMS depth profiles of $^{28}$Si$^+$, $^{16}$O$^+$, $^{11}$In$^+$, $^{28}$Si$^{16}$O$^+$, and $^{90}$Zr$^+$ in (a) N$_2$ and (b) O$_2$ PDA samples before, i.e., fresh MOS capacitors, and after formation of conductive paths, i.e., from $V_g = -20$ V, 20 min stress. Since the size of the SIMS spot is large, i.e., 50 μm x 50 μm, signals in the SSI-LED are contributed by both conductive paths and non-conductive path areas. For the N$_2$ PDA SSI-LED, $^{28}$Si$^+$ is detected in the ITO layer after the stress, which indicates the diffusion of the substrate material through the conductive paths to the gate electrode.

The similar phenomenon of Si atoms migration from the substrate to the gate electrode during the dielectric breakdown of a MOSFET was observed. There are no major changes in $^{11}$In$^+$, $^{16}$O$^+$, $^{90}$Zr$^+$, and $^{28}$Si$^{16}$O$^+$ signal distributions before and after the $V_{BD}$ stress in the N$_2$ PDA samples. The intensity of $^{28}$Si$^+$ in the ITO film of the O$_2$ PDA SSI-LED is 15 times larger than that in the original MOS capacitor. It is also much larger than that in the N$_2$ PDA SSI-LED, which can be explained by the higher local temperature of the conductive path in the O$_2$ PDA SSI-LED sample. After the stress, the O$_2$ PDA sample has broad tails of the $^{28}$Si$^{16}$O$^+$, $^{16}$O$^+$, $^{11}$In$^+$, $^{28}$Si$^{16}$O$^+$, and $^{90}$Zr$^+$ signals, which can also be contributed by the high temperature of the
Figure 10. SIMS profiles of $^{28}\text{Si}^+$, $^{16}\text{O}^+$, $^{115}\text{In}^+$, $^{28}\text{Si}^{16}\text{O}^+$, and $^{90}\text{Zr}^+$ of (a) N$_2$ and (b) O$_2$ PDA samples pre- (fresh capacitors) and post-stress (after breakdown with $V_g = -20$ V for 20 min).
The higher temperature of the conductive path in the O₂ PDA SSI-LED is also responsible for its higher light emission efficiency.

Conclusions

Electrical and light emission characteristics of SSI-LEDs made from MOS capacitors of the same sputter-deposited ZrHIO high-κ film but different PDA atmospheres have been studied. The O₂ PDA capacitor contains a thicker and more SiOₓ-rich HfSiOₓ interfacial layer as well as lower defect densities than those of the N₂ annealed capacitor. Therefore, the former has a lower leakage current and a higher dielectric breakdown strength, which makes it more difficult for a capacitor to form conductive paths. When stressed at the same voltage, conductive paths in the O₂ PDA SSI-LED are more effectively excited to emit light stronger than those in the N₂ PDA SSI-LED. The CIE chromaticity coordinates, CRI’s, and CCT’s of the N₂ and O₂ PDA SSI-LEDs are close to those of the conventional incandescent lamp. The emission spectrum of the O₂ PDA SSI-LED is slightly more bluish than that of the N₂ PDA SSI-LED because of the higher local temperature in the conductive path. The SIMS elemental analysis result shows higher concentrations of Si and O in the high-κ stack of the O₂ PDA SSI-LED probably due to the faster diffusion of these elements through the conductive path. In summary, the PDA atmosphere affects the high-κ stack’s physical and material properties in the original capacitor, which influences the formation mechanism, composition, and number of conductive paths and eventually the electrical and optical characteristics of the SSI-LED.

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