Back-End-of-Line Compatible Low-Temperature Furnace Anneal for Ferroelectric Hafnium Zirconium Oxide Formation

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The discovery of ferroelectricity in thin doped hafnium oxide films revived the interest in ferroelectric (FE) memory concepts. Zirconium-doped hafnium oxide (HZO) crystallizes at low temperatures (e.g., 400 °C), which makes this material interesting for the implementation of FE functionalities into the back end of line (BEoL). So far, the FE phase of prior amorphous HZO films is achieved by using a dedicated rapid thermal annealing (RTA) treatment. However, herein, it is shown that this dedicated anneal is not needed. A sole furnace treatment given by the thermal budget present during the interconnect formation is sufficient to functionalize even ultrathin 5 nm HZO films. This result helps to optimize the integration sequence of HZO films (e.g., involving a minimum number of BEoL process steps), which saves process time and fabrication costs. Herein, metal–FE–metal capacitors with Hf_{0.5}Zr_{0.5}O_{2} films of different thicknesses (5–20 nm) are fabricated annealed at 400 °C for various durations within different types of ovens (RTA and furnace). Structural and electrical characterization confirms that all furnace-annealed samples have similar X-ray diffraction patterns, remanent polarization, endurances, and thickness dependencies as RTA-annealed ones. With respect to remanent polarization, leakage current, and endurance, the HZO film of 10 nm thickness shows the most promising results for the integration into the BEoL.

1. Introduction

Nonvolatile memory concepts based on conventional perovskite-type ferroelectrics (FEs) like lead zirconate titanate and barium titanate were proposed already in the late 1950s.[1] However, their challenging integration due to incompatibility with complementary metal-oxide–semiconductor (CMOS) technologies has limited their use to niche applications and restricted scaling below the 90 nm node. This is particularly because the conventional FEs require high film thicknesses, high annealing temperatures, and provide rather small coercive and breakdown fields as well as high dielectric constants.[2] The discovery of ferroelectricity in HfO_{2},[3] a material that is successfully introduced into standard CMOS high-k metal gate technologies,[4] has led to a renaissance of FE memory concepts.[5] The ferroelectricity in HfO_{2}-based films is attributed to the formation of a polar, noncentrosymmetric, orthorhombic (o) phase with the space group Pca2_{1}.[6] This thermodynamically unstable phase can be stabilized by introducing several dopants like silicon,[7] zirconium,[8] gadolinium,[9] yttrium,[10] and aluminum[10] into thin HfO_{2} films.

The integration of FE memory concepts into the back end of line (BEoL) of modern integrated circuits offers several benefits: 1) The effective memory area of the chip can be strongly increased (i.e., more functionalities can be added). 2) FE nonvolatile memory concepts like FE random-access memories (FeRAMs), nonvolatile static RAM (nvSRAM), and metal–FE–metal–insulator–semiconductor (MFMIS)-based FE field-effect transistors (FeFETs) can be added to the chip without introducing major changes in the front-end-of-line (FEoL) design. 3) Due to the symmetric electric field condition, the metal–FE–metal (MFM) capacitors BEoL integration offers an improved endurance (∼10^{5} cycles) compared with FeFETs (∼10^{5} cycles).[11]

Among the dopants mentioned previously, zirconium is the most promising for the integration of FE functionalities into the BEoL. This is mainly due to two reasons: 1) The system Hf_{x}Zr_{1−x}O_{2} (HZO) shows the FE phase over a wide range of concentrations x with the maximum remanent polarization P_r at around x = 0.5.[8] This ratio can be easily realized using common deposition techniques like atomic layer deposition (ALD) with a homogenous concentration throughout the film. 2) The FE properties of HZO films can be achieved at low process temperatures...
of about 400 °C. This is most likely due to the low crystallization temperature of ZrO₂ compared with other potential high-k oxides. Such a peak temperature is compatible to state-of-the-art BEOL processing, where higher annealing and/or process temperatures will lead to an undesired diffusion of metal atoms.

It was shown that thin HZO films could be crystallized successfully in the desired FE phase by applying rapid thermal annealing (RTA) at 400 °C and even 300 °C for 30 or 60 s. Furthermore, phase transition kinetics were studied (at prior RTA crystallized films) during supplementary annealing at different temperatures and durations to simulate the thermal profile present during the formation of the interconnects, where high-temperature processes (up to 400 °C) occur several times and last from some minutes to hours.

However, the effect of a sole furnace anneal on the crystal structure and FE properties of HZO films is not reported so far. Without the need of a dedicated RTA crystallization anneal, the thermal budget present during the interconnect formation could be used to “in situ” crystallize the HZO films, which saves process time and fabrication costs. To find an optimal integration sequence (e.g., involving a minimum number of BEOL process steps), different anneal conditions (RTA vs furnace) and a wide range of annealing times (60 s, 1 h, and 2 h) are investigated with respect to the FE, electrical, and structural properties of Hf₀.₅Zr₀.₅O₂ films embedded in MFM capacitors.

2. Experimental Section

The MFM capacitors (cf., Figure 1) were fabricated on highly boron-doped 300 nm silicon wafers. The FE HZO layers were deposited via ALD (ASM Polygon 8300 equipped with a Pulsar 3000 chamber) at a temperature of 300 °C. As precursors, hafnium tetrachloride (HfCl₄) and zirconium tetrachloride (ZrCl₄) were used together with water (H₂O) as the oxidizing reactant and argon (Ar) as purging gas. The constant film stoichiometry (Hf₀.₅Zr₀.₅O₂) was achieved using a precursor cycling ratio of 1:1. The film thickness was varied between 5 and 20 nm, using a different number of total deposition cycles and confirmed by spectroscopic ellipsometry (KLA Tencor Spectra FX100).

The TiN bottom electrode was deposited by ALD in an ASM A412 batch process chamber at a temperature of 450 °C using TiCl₄ and NH₃ precursors. The TiN top electrode was fabricated by magnetron sputtering at room temperature to avoid in situ crystallization of the FE.

The HZO crystallization anneal was done at a temperature of 400 °C in N₂ atmosphere. Two oven types were used: 1) RTA (Mattson Helios XP) with a hold time of 60 s, a ramp up rate of 50 K s⁻¹, and passive cool down and 2) a furnace (ASM A412) for a duration of 1 or 2 h. In the latter case, the wafers were introduced into a prior heated-up chamber and pulled out after processing.

The MFM stack was visually analyzed (Figure 1) on cross-section specimen using a FEI Tecnai F20 transmission electron microscope (TEM). The structural characteristics of the crystalline phases were concluded from X-ray powder diffraction experiments in a glancing-angle diffraction geometry (GAXRD) with a thin-film diffractometer (Bruker D8 Discover). To avoid the substrate reflection 311, all samples were mounted with a 45° rotation between the [001] direction of the (100)-oriented silicon wafer and the diffraction plane.

For the electrical characterization, Ti/Pt dots were patterned by applying shadow mask and electron beam evaporation. Subsequently, TiN between the dot contacts was removed by a wet etching step (SC1) to form individual MFM capacitors. The polarization-field (P–E) measurements were carried out with an Aixacct TF 3000 FE analyzer using a triangular waveform at a frequency of 1 kHz. The endurance tests were performed with an electrical cycling frequency of 10 kHz.

3. Results and Discussion

A thermal budget of 400 °C and 60 s (RTA anneal) is sufficient to crystallize all tested HZO films with thicknesses of 10 nm and above (cf., Figure 2). Only in the case of the 5 nm thin layer, a longer annealing time (e.g., furnace anneal of 1 h) is required to trigger crystallization.

Using XRD, the monoclinic (m)-phase can be distinguished from the cubic (c)-, tetragonal (t)-, and o-phase. Interestingly, the anneal duration (60 s vs 1 h) and the oven type (RTA vs furnace) have only minor impact on the formed fraction of the m-phase. In the case of the 10 nm film, the impact is negligible. However, all 10 nm films annealed in the furnace are slightly more crystalline than the ones annealed in RTA. In the case of the 15 and 20 nm films, the amount of the m-phase is slightly higher, when annealing occurs in the furnace.

Nevertheless, a clear correlation between the fraction of the m-phase and the film thickness can be seen, which is—with the exception of the 5 nm films—indeed of the used oven and annealing time; in the case of the 5 nm films, no diffraction lines of the m-phase are visible. At 10 nm film thickness, first diffraction lines appear (cf., the most pronounced line at 2θ ≈ 28° in Figure 2). The intensity of these lines increases with the film thickness. It is known that the fraction of the m-phase increases with film thickness. However, usually reasonable amounts of this phase are detected only for films exceeding 20 nm. The reason for detection here at 20 nm could be the partial crystallization of...
the films during the deposition process. It is known that a TiN capping layer, which applies tensile stress to HZO, is beneficial during annealing to achieve the FE o-phase.\textsuperscript{[15]} Because this capping is missing for “in situ” crystallized films, the m-phase is more pronounced in this case.

From XRD, it is not straightforward to distinguish between the c-phase, t-phase, and o-phase, because all mentioned phases have reflection lines at similar 2θ angles. Therefore, and to attain an understanding of the FE-film properties, electrical measurements were conducted (Figure 3). An important result is that for 10–20 nm films, the shape of the $P–E$ curves looks very similar for all investigated annealing durations and oven types. However, again a thickness dependence can be seen: The 5 nm thin film annealed with RTA (60 s) shows dielectric behavior in the pristine state (cf., Figure 3a) and after $10^5$ wake-up cycles (cf., Figure 3e), which is in good agreement with the amorphous state of the material. Films of the same thickness, but furnace annealed for 1 and 2 h, depict antiferroelectric (AFE)-like behavior in the pristine state (cf., Figure 3a). Therefore, the 5 nm films crystallize primarily in the AFE-like t-phase. With increasing film thickness, the AFE t-phase gradually vanishes. Although the 10 nm HZO films show a joint AFE (i.e., t-phase) and FE (i.e., o-phase) behavior (cf., Figure 3b), no pinching can be found for the thicker films (cf., Figure 3c,d).

For pure ZrO\textsubscript{2} films, the t-phase is energetically favorable at small crystallite sizes due to the surface energy effect.\textsuperscript{[23]} In the case of the 5 and 10 nm films, such a small crystallite size is enforced by film thickness. As this thickness increases, a phase transformation occurs from the t-phase to the m-phase. This can be seen from GAXRD and the shape of the $P–E$ curves (cf., Figure 3b–d). Consequently, to achieve the largest share of the o-phase, a medium-layer thickness between 10 and 15 nm is the most promising.

During wake-up, the t-phase (present for thin films at the pristine state) vanishes gradually with the number of applied field cycles. After $10^5$ cycles, this process is finished (cf., Figure 3e,f). This suggests the occurrence of an initial field-induced phase transformation from a t-rich state to an o-rich state\textsuperscript{[23]} with a subsequent stabilization of the o-phase due to defect migration and/or domain depinning. For thicker films, the wake-up process has a smaller effect on the shape of the $P–E$ curves. This is due to the smaller fraction of the t-phase in this case.

Another important parameter of the HZO films is the leakage current, which leads to a “cigar-like” hysteresis loop and, therefore, can be estimated qualitatively from its shape. In particular, after wake-up cycling, a clear trend is visible: For the same field, thicker films encounter higher leakage than thinner ones (cf., Figure 3e–h). Consistent with this observation is that the breakdown field also decreases with film thickness. Therefore, a limited field of 2.5 MV cm$^{-1}$ was applied to the 20 nm-thick film (Figure 3d,h), whereas for all other thicknesses up to 3.0 MV cm$^{-1}$ could be applied. A possible reason for this thickness-dependent degradation could be that thicker films have a lower share of the amorphous phase and/or consist of larger crystallites. Both facilitate the diffusion/field drift of the charges along the grain boundaries, leading to increased leakage and dielectric breakdown.\textsuperscript{[24]}

With the exception of the 5 nm films, where only furnace annealing triggered crystallization of the FE film, the duration (60 s–2 h) and the anneal method (RTA vs furnace) have only a minor impact on $P_c$ (cf., Figure 4). In the case of 10 nm films, $P_c$ is marginally higher for furnace-annealed samples than for samples annealed with RTA. This is in good agreement with the slightly better crystalinity observed for films annealed in the furnace (cf., Figure 2). In the case of 15 and 20 nm films, $P_c$ decreases slightly with the annealing duration. This is in good agreement with the increased fraction of the m-phase formed for longer annealing times. Nevertheless, annealing times up to 2 h will not degrade the HZO films significantly. Consequently, the thermal budget during the formation of interconnections will not harm the FE properties and, furthermore, can be used for its crystallization. A dedicated RTA anneal is then not required.

However, $P_c$ depends strongly on the film thickness (cf., Figure 4). The measured $P_c$ value 1) increases initially between 5 and 10 nm, 2) remains approximately constant between 10 and 15 nm, and 3) decreases again for films of 20 nm. This thickness dependence is attributed to the partial formation of the t-phase and m-phase, which occurs during annealing of rather thin or thick films, respectively (cf., Figure 2 and 3). In addition, for
Figure 3. Polarization versus electric field measurements of MFM capacitors. The HZO films have thicknesses ranging from 5 to 20 nm and were annealed at 400 °C either using RTA (60 s) or a furnace-type oven (1 or 2 h). The hysteresis loops were plotted for a–d) the virgin state and e–h) after $10^5$ cycles.

Figure 4. Remanent polarization of MFM capacitors after wake-up. The HZO films have different thicknesses: a) 5 nm, b) 10 nm, c) 15 nm, and d) 20 nm. Annealing was performed under different conditions: 1) RTA for 60 s, 2) furnace for 1 h, and 3) furnace for 2 h. Each box plot consists of 40 measurements. For films below 20 nm, a maximum field of 3.0 MV cm$^{-1}$ was used. At 20 nm film thickness, a limited field of 2.8 MV cm$^{-1}$ was used due to the weaker electrical robustness.
thinner films (e.g., 5 nm), the fraction of amorphous residues may be greater due to the surface energy effect, which can be estimated from the intensities of the diffraction lines in Figure 2. A similar size dependence was observed by Kim et al. for RTA-annealed (60 s at 400 °C) HZO films.\(^{[13]}\)

Consequently, the largest \(P_r\) values of 19 \(\mu\text{C cm}^{-2}\) (10 nm) and 20 \(\mu\text{C cm}^{-2}\) (15 nm) are measured for films of medium thicknesses. However, as mentioned before, the 15 nm film encounters a higher leakage (cf., Figure 3g). Hence, the HZO films of 10 nm are the most promising for an integration into the BEoL. Nevertheless, films of 5 nm thickness have the advantage of a considerable high \(P_r\) value (≈10 \(\mu\text{C cm}^{-2}\)) at a low operation voltage of 1.5 V.

The endurance in Figure 5 can be divided into two stages: wake up and fatigue. During the wake-up process, \(2P_r\) increases (i.e., a depinching of the pristine pinched hysteresis loop occurs) with the number of field cycles. This stage is attributed to a gradual decrease in the build-in bias field due to a redistribution of defects (oxygen vacancies) and/or a partial phase transition of the dielectric material from the t-phase to the o-phase.\(^{[25]}\)

During fatigue (or aging), \(2P_r\) decreases again due to the generation of new defects like oxygen vacancies, which facilitate charge trapping and/or domain pinning.\(^{[12,25]}\)

The maximum number of field cycles used for the endurance tests was \(10^7\) (cf., Figure 5). Remarkably, the 5 nm HZO film survived the whole cycling test at 3 MV cm\(^{-1}\) without dielectric breakdown. At the same field, the dielectric breakdown occurred after \(8 \times 10^5\) and \(10^5\) cycles for the FE films of 10 and 15 nm, respectively. Furthermore, in case of the 20 nm film, no cycling experiments could be done at 3 MV cm\(^{-1}\), due to initial dielectric breakdown. This strong-size dependence of the breakdown field is due to different reasons: 1) Thicker films have statistically a higher probability for defects. Because the field is constant for all thicknesses, the occurrence of defects will inevitably reduce the life time of the device. 2) Due to the surface energy effect, thicker layers are expected to have a higher crystalline-to-amorphous volume fraction than thinner ones. It is known that rather amorphous films have a superior dielectric quality.\(^{[24]}\) This effect can also be seen by the increasing leakage current with the film thickness in Figure 3. 3) The thicker films have a greater fraction of the m-phase (cf., Figure 2). Compared with the other crystalline modifications, the \(k\) values of this phase are rather small.\(^{[26]}\) This, in turn, changes the field distribution across the FE film, which can negatively affect the endurance. It was reported in the literature that the m-phase can work as a "dead layer" in FEs and can thus degrade the FE properties and endurance of HZO capacitors.\(^{[17,27]}\)

The number of cycles until dielectric breakdown decreases with increasing electrical field applied to the MFM capacitors (cf., Figure 6). For the studied range between 2 and 4 MV cm\(^{-1}\), this dependency is exponential (at a first approximation). Thus, the data points in Figure 6 can be fitted by a straight line to estimate the endurance for certain electrical fields. At 5 nm

![Figure 5. Endurance of HZO films of different thicknesses (5–20 nm) stressed at electrical fields between 2 and 4 MV cm\(^{-1}\). Annealing was performed at 400 °C for 1 h within furnace oven.](image-url)

![Figure 6. Endurance versus electrical field for MFM capacitors with different HZO thicknesses ranging from 5 to 20 nm. Annealing was performed at 400 °C within an RTA tool (60 s, open square) or within a furnace oven for 1 h (open stars) or 2 h (double cross). The maximum number of field cycles was \(10^7\). Devices with data points in brackets passed the cycling test. The (on a first approximation) exponential field dependence of the endurance was used to plot straight lines.](image-url)
thickness, an endurance of $4 \times 10^7$ cycles was estimated for the furnace-annealed samples. This is comparable with the result obtained by Kim et al. (about $10^8 \times 2.6 \text{MV cm}^{-1}$) for HZO films of the same thickness but annealed at 450 °C with RTA.\(^\text{[12]}\) Also for all other data points in Figure 6, no clear impact of the duration (60 s to 2 h) and the oven type (RTA and furnace) on endurance can be found.

In conclusion, MFM capacitors with Hf$_{0.5}$Zr$_{0.5}$O$_2$ films of different thicknesses (5–20 nm) were fabricated and annealed at 400 °C. Annealing was performed either within an RTA tool for 60 s or within a furnace oven for 1 or 2 h. It was shown that a sole furnace treatment of 1 h is sufficient to crystallize even ultrathin films of 5 nm thickness partly in the FE o-phase. Furthermore, all furnace-annealed samples had similar $P_r$, values, endurance, and thickness dependencies as RTA-annealed ones. Thus, to involve a minimum number of BEOL process steps, the thermal budget of the interconnect formation can be used to “in-situ” crystallize the HZO films without the need of a dedicated RTA anneal. With respect to $P_r$, leakage current, and endurance, the HZO film of 10 nm thickness shows the most promising results for the integration into the BEOL.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

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