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Microelectronics Reliability 45 (2005) 819–822
MICROELECTRONICS RELIABILITY
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Electrical properties of hafnium silicate films obtained from a single-source MOCVD precursor

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Received 28 June 2004; received in revised form 3 September 2004
Available online 25 December 2004

Abstract

In the present work, the potential of hafnium silicate ($Hf_xSi_yO_2$) films as an alternative gate dielectric to SiO_2 for future technology generations is demonstrated. Thermally stable $Hf_xSi_yO_2$ films are deposited from a single-source MOCVD precursor. *I–V* and *C–V* measurement data are presented and effects of post-deposition annealing on electrical properties are discussed. A 900 °C O_2-anneal shows best results in terms of leakage current characteristics and is, therefore, intensively investigated.
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1. Introduction

The aggressive thickness scaling of SiO_2 in complementary metal-oxide-semiconductor (CMOS) processing causes several limitations, such as high leakage currents, enhanced boron penetration, and reliability limitations [1]. Therefore, new high permittivity (high-*k*) gate dielectrics to replace SiO_2-based dielectrics in future technology generations are urgently needed [1]. Intensive studies in the last years revealed ZrO_2 and HfO_2 as promising candidates. Though, the permittivity of silicates and aluminates is lower compared to their respective oxides, Zr and Hf silicates and aluminates are also widely investigated [1–4]. Si or Al are incorporated to increase the crystallization temperature, to reduce oxygen conductivity, and to improve the interface properties to the Si substrate [1]. In this paper, hafnium silicate films ($Hf_xSi_yO_2$) deposited by metal-organic chemical vapor deposition (MOCVD) from a single-source precursor are investigated. We present results of capacitance–voltage (*C–V*) and current–voltage (*I–V*) measurements and discuss influence of post-deposition annealing on electrical properties and current conduction mechanisms through the films.

2. Sample preparation

Electrical characterization was performed using metal insulator semiconductor (MIS) capacitors manufactured on 5 Ω cm p-type Si wafers. After surface conditioning in diluted HF, the $Hf_xSi_yO_2$ films were deposited immediately by MOCVD using a single-source precursor

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0026-2714/$ - see front matter © 2004 Elsevier Ltd. All rights reserved.
doi:10.1016/j.microrel.2004.11.040

(Hf(acac)$_2$(OSi^{t}BuMe$_2$)$_2$). The synthesis, structure, and characterization of an equivalent Zr precursor were described in a recent publication [2]. The depositions are performed at 550 °C in Ar/O$_2$ atmosphere at a total pressure of 2.5 mbar. A part of the samples receive a rapid thermal annealing (RTA) for 10 s in O$_2$ or N$_2$ ambient, respectively, at temperatures ranging from 700 to 900 °C. Finally, gate electrodes consisting of stacked 20 nm Ni and 500 nm Al with an area of 2–4 × 10^4 cm^2 are evaporated through a shadow mask.

3. Results and discussion

C–V characteristics are measured at frequencies, f_m, ranging from 10 to 1000 kHz on the basis of a two-element parallel model. The measured C_m–V and D_m–V curves are shown in Fig. 1a (capacitance C_m, dissipation D_m equal $(\omega C_m G_m)^{-1}$, parallel conductance G_m). A considerable discrepancy, both, in accumulation and flat band region is seen ruling out an accurate analysis of the obtained data. The reason for this change in *C–V* characteristics with f_m is the lack of accuracy of a two-element circuit model when measuring thin and/or leaky dielectrics. Using the method presented by Yang and Hu [5], the model can be numerically expanded to a more precise three-element model, which takes also the series resistance into account. Performing the correction according

$$C = \frac{f_{m1}^2 C_{m1}(1 + D_{m1}^2) - f_{m2}^2 C_{m2}(1 + D_{m2}^2)}{f_{m1}^2 - f_{m2}^2}, \qquad (1)$$

(indices 1, 2 represent two different f_m) unique *C–V* traces are obtained as shown in Fig. 1a, too. Fig. 1b shows the corrected *C–V* characteristics for an as-deposited film and samples annealed under different RTA conditions. Table 1 summarizes the results. For the permittivity, k, no clear trend is noticeable, e.g. the values are about 12. However, the physical thickness, d_{phys}, measured by ellipsometry and also the equivalent oxide thickness, EOT, determined by *C–V* change with different RTA conditions. Similar results, i.e. the decrease of d_{phys} of about 20% due to an O$_2$-RTA step, were found for Zr silicate films deposited from an equivalent precursor [3]. Film analysis by X-ray photoelectron spectroscopy (XPS) reveals that for as-deposited and N$_2$-annealed films, the C contamination is up to 20 at.%. For samples annealed in O$_2$ atmosphere, no C is found in the films within the detection limit of XPS. Therefore, a possible explanation for the loss of physical thickness is the C elimination due to O$_2$-annealing. In terms of flatband voltage, V_{FB}, and (fixed) effective charge, Q_{eff}, respectively, the values differ substantially depending on post-deposition RTA (Table 1). For the calculation of Q_{eff}, a workfunction difference, Φ_{MS}, of −0.5 V is taken into account. For the as-deposited film, a negative fixed charge is detected, which increases after the N$_2$-RTA step. As both samples show a considerable high C contamination level, a part of this negative charge can be related to C. For the N$_2$-annealed sample, no N is detected in the film by XPS. Hence, we assume that a N$_2$-RTA degrades the bonding structure within the dielectric, e.g. more dangling bonds and correspondingly negative charges exist. This is also supported by *J–V* measurements (Fig. 2a), where this sample shows highest leakage currents. In the case of the O$_2$-annealed samples, the C-related negative charge is eliminated. However, a negative charge is still detected after the 700 °C O$_2$-RTA. By increasing the annealing tempera-

Table 1
Parameters extracted from corrected *C–V* characteristics for samples processed with different RTA steps

RTA	d_{phys} (nm)	EOT (nm)	k	V_{FB} (V)	Q_{eff} (10^{12} cm^{-2})
w/o	14.6	5.2	11.0	0.0	−2.1
N$_2$/700 °C	13.0	4.1	12.4	0.4	−4.8
O$_2$/700 °C	11.0	3.8	11.3	−0.3	−1.1
O$_2$/900 °C	10.5	3.3	12.4	−1.6	7.2

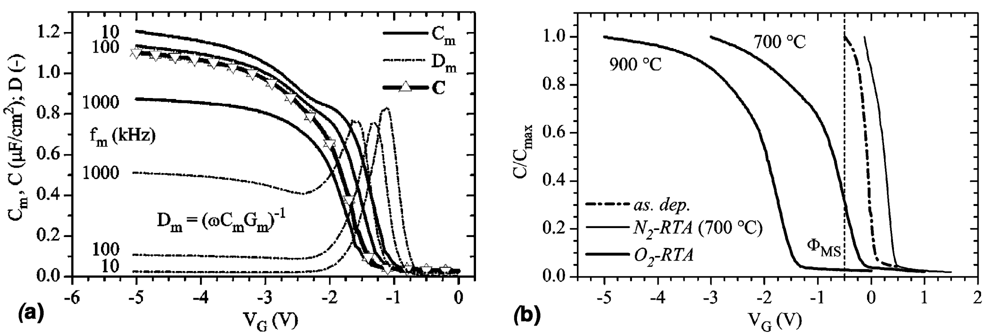

Fig. 1. (a) Measured C_m–V and D_m–V curves at different frequencies and corrected *C–V* characteristic for 900 °C O$_2$-annealed sample. (b) Corrected *C–V* curves for as-deposited and under different conditions annealed films.

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