

# Samarium oxide thin films deposited by reactive sputtering: Effects of sputtering power and substrate temperature on microstructure, morphology and electrical properties

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## ABSTRACT

In this study, the effects of increase in sputtering power and substrate temperature on the microstructural, morphological and electrical characteristics of Sm<sub>2</sub>O<sub>3</sub> thin films have been reported. All films were deposited at 50 °C with varying in sputtering power from 100 to 250 W in 50 W steps by the reactive sputtering technique. The crystallization, grain size, and d-spacing of the films were determined by an X-ray diffractometer (XRD). The semi-quantitative compositional changes were investigated by energy-dispersive X-ray (EDX) spectroscopy while microstructural and morphological modifications were studied by atomic force microscopy (AFM). In addition, the electrical evolutions of the films were determined by alternating current (a.c.) conductivity. It was observed that the crystallization of the films was increased with increase in sputtering power up to 200 W and decreased at the power of 250 W. Therefore, the films deposited at 200 W were annealed at 50 °C, 125 °C, and 200 °C, and a continuous crystallization improvement was observed. The composition of the films was improved by both sputtering power and increase in substrate temperature. The grain boundaries and surface roughness of the films were also found to be significantly affected by the change in substrate temperature. The a.c. conductivity of the films gradually decreases from  $1.10 \times 10^8 \Omega^{-1} \text{cm}^{-1}$  to  $2.50 \times 10^6 \Omega^{-1} \text{cm}^{-1}$  by increase in sputtering power and substrate temperature. The results show that the Sm<sub>2</sub>O<sub>3</sub> thin films fabricated at 200 W sputtering power and 200 °C substrate temperature exhibit requested structural and morphological characteristics.

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## 1. Introduction

Owing to the continuous downscaling of the complementary metal-oxide-semiconductor (CMOS) devices, the investigations of finding new alternative high-dielectric constant (high-*k*) materials have gained great interest in the last decades [1]. Various candidates for high-*k* dielectrics, e.g. HfO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and ZrO<sub>2</sub>, have been studied during the

past decades [2–6]. However, several issues may arise from combining these high-*k* materials with the Si substrates, the major problem being the instability of the interface between high-*k* metal oxide and the underlying Si. Quite often unfavorable SiO<sub>x</sub> and/or silicate phases, which may degrade device performance, have been observed after high temperature film deposition process and/or annealing treatment of amorphous high-*k* oxides [7–9]. To solve these problems, rare-earth oxides [10–13] have been investigated as promising gate dielectric candidates for future MOS-device applications [11,13–15]. The rare-earth oxides are predicted to be stable in contact with the Si up to 1000 °C, preventing the formation of silicide layers [11]. Among other rare-oxides,

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samarium oxide ( $\text{Sm}_2\text{O}_3$ ) has some basic attractive characteristics such as high dielectric constant (5.25–30 [10,14] depending on thickness and film quality), wide band gap [16], large conduction band offsets [17], thermodynamic stability on the underlying Si surface [18,19], and low hygroscopic characteristics.

Various deposition methods, such as metal-organic chemical vapor deposition (MOCVD) [20], pulsed laser deposition (PLD) [21], electron beam evaporation [22], and sputtering [10], can be used to grow the  $\text{Sm}_2\text{O}_3$  thin films. Out of these methods, sputtering has some advantages. A lower deposition temperature eliminates formation of impurity phases in the Si layer, i.e.  $\text{SiO}_x$ . Moreover, sputtering can be easily scaled up from small-sized laboratory targets to large-scale industrial machines and provide uniformity of the films, good reproducibility of film properties, and a simple deposition process [18].

Compositions and structural quality of thin films are directly related to deposition parameters [23–25] in the sputtering methods and any change in structural parameters may affect electrical characteristics of the devices. The thin films having good crystallinity and larger grain size are required in electronic applications owing to the reduced influences from grain boundaries and defects densities in the structure. Therefore, in this work we aim to specify an optimum fabrication process to obtain good crystallinity in  $\text{Sm}_2\text{O}_3$  thin films. To do this, we studied the possible effects of the various sputtering powers,  $P_w$ , and substrate temperatures,  $T_s$ , on the microstructural and morphological characteristics of the  $\text{Sm}_2\text{O}_3$  films fabricated by a reactive radio frequency (RF) sputtering method. The effects of these parameters on electrical characteristics were also investigated by determining a.c. conductivity of the films by capacitance and conductance measurements. The crystallinities of the films were investigated by an X-ray diffractometer (XRD), while the compositional changes on the film were analyzed by energy-dispersive X-ray (EDX) spectroscopy. Microstructural, morphological characteristics and height asymmetries of films were studied by atomic force microscopy (AFM).

## 2. Experimental details

The  $\text{Sm}_2\text{O}_3$  thin films were deposited on n-type (100) Si substrate with a resistivity of 1–4  $\Omega$  cm by reactive RF sputtering from a 4-in. samarium target with purity of 99.99%. Before the deposition of  $\text{Sm}_2\text{O}_3$  layers, the wafers were cleaned with standard RCA cleaning process. RF magnetron sputtering was carried out in an ultra-pure Ar (99.9999%) and  $\text{O}_2$  (99.9999%) mixed gaseous atmosphere by supplying RF power at a frequency of 13.56 MHz. The flow rates of Ar and  $\text{O}_2$  were controlled by a flow meter (HELIOS, CS 200). The ideal flow rates from the literature [26] were used: 15 sccm for Ar and 10 sccm for  $\text{O}_2$ . For all films, the target–substrate distances were kept constant at 100 mm. Prior to commencing sputtering, the base pressure of the chamber was below  $4.0 \times 10^{-4}$  Pa and sputtering gas pressure was 1.0 Pa. In addition, pre-sputtering was done for 10 min to remove any impurities present on the target surface. Before the sputtering process, cleaned n-Si wafers were divided into two groups in order to study two different sputtering parameter effects. In the first group, wafers were deposited at various sputtering

powers (100 W, 150 W, 200 W, and 250 W) at 50 °C, followed by annealing at 800 °C for 40 min in Nitrogen environment.

To examine the degree of crystallinity of the first group of samples, XRD measurements were performed in the range of ( $2\theta=$ ) 25–65° with  $\text{CuK}\alpha$  radiation ( $\lambda=1.5418$  Å). After considering the XRD analysis results of the first group samples, the second group of the films was deposited at various substrate temperatures (50 °C, 125 °C and 200 °C) at 200 W sputtering power only. These samples were also annealed at 800 °C for 40 min in Nitrogen environment. Then, the XRD measurements were performed under the same conditions as for the first group of samples. The compositional changes for all fabricated films were investigated by EDX and AFM measurements to characterize the microstructure and surface morphology of the films.

For electrical measurements, aluminum (Al) contacts were obtained: back ohmic contacts were deposited via sputtering on the fabricated samples and front electrodes were formed in circular dots of 2.5 mm diameter through a shadow mask. To determine a.c. conductivity of the films, capacitance and conductance measurements were performed at 1 MHz at the strong accumulation gate voltage of +3.0 V.

## 3. Results and discussions

### 3.1. X-ray diffraction (XRD) results

In this work, the changes in crystallinity and phase evolution of  $\text{Sm}_2\text{O}_3$  thin films have been studied by X-ray

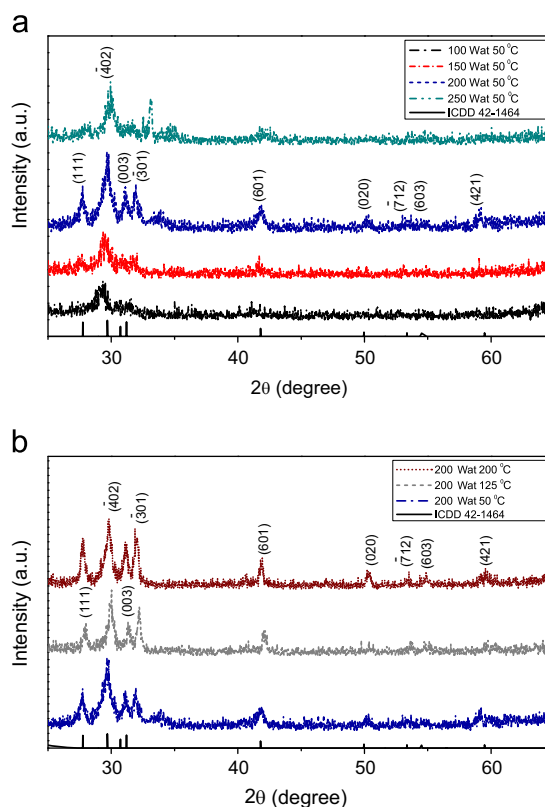


Fig. 1. XRD spectra of the  $\text{Sm}_2\text{O}_3$  thin films deposited at various (a) RF powers and (b) substrate temperatures.

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