Preparation and Characterization of Sol-Gel Derived High-k SrTa₂O₆ Thin Films

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Abstract: SrTa₂O₆ (STA) thin films were fabricated by sol-gel method. The films annealed below 700 ℃, showed amorphous phase and crystallization phase was observed after annealing over 800 ℃. From high frequency capacitance-voltage measurements, 24nm thick STA thin film annealed at 900 ℃, has an EOT of 5.7nm and a dielectric constant of 16. Leakage current characteristics were improved by the insertion of chemical oxide between STA and Si. Leakage current densities are around 3.5×10⁻⁷A/cm² at 5V for the structure inserted chemical oxide but 1.4×10⁻⁶A/cm² at 5V without chemical oxide.

Key Words: SrTa₂O₆, STA, strontium tantalates, high-k, sol-gel, chemical oxide

1. Introduction

Silicon dioxide thin films have been commonly used as dielectric materials for gates and capacitors in VLSI. The rapid progress of the IC technology requires a gate dielectric below 2nm[1]. However, continuous scaling of gate oxide has run into difficulties due to high direct tunneling leakage current[2].

To solve these problems, as an alternative to silicon oxide systems, high-*k* metal oxide thin films have been investigated[1-3]. Higher permittivity means that thicker dielectric can be available and, thereby leakage current and capacitance can be improved. The most commonly studied high-*k* materials include Ta₂O₅, SrTiO₃, Al₂O₃, HfO₂, and ZrO₂, which have dielectric constants ranging from 10 to 80[1, 4]. Recently, it was reported that SrTa₂O₆ (STA) thin films deposited by MOCVD or ALCVD, have a dielectric constant up to 40 - 100 depending on its crystalline form and low leakage current density[5, 6].

In this study, we prepare STA thin films on silicon substrates using sol-gel method. The electrical properties of the metal-insulator-silicon (MIS) structure are investigated with C-V and I-V characteristics at various annealing conditions and thickness of thin film.

2. Experiments

STA thin films were prepared on p-type (100) silicon substrates by sol-gel. The STA was spin-coated at 5000rpm for 20s and then pre-baked at 350°C for 10min. Two types of substrates were prepared: HF-last and chemical oxidized silicon. Chemically oxidized silicon were obtained by dipping substrate in hydrogen peroxide (H_2O_2) solution for 30min after removing native oxide with BOE (buffered oxide etchant). The pre-baked films were annealed by rapid thermal annealing at temperature ranging from 600 to 900°C in O_2 atmosphere. To determine the electrical properties, electrodes of Au were deposited by thermal evaporation through shadow mask on top and bottom of the STA/Si structures.

C-V and I-V characteristics of the films with Au/STA/Si/Au

structure, were tested by HP4280A and HP4156C. The structural properties and morphology of the films were characterized by XRD(X-ray diffraction) and AFM(atomic force microscopy).

3. Results and Discussion

Figure 1 (a) shows X-ray diffraction patterns of STA thin films annealed at different temperatures in O_2 . Below $700\,^{\circ}$ C, there were no recognizable diffraction peaks, implying an amorphous structure. Several peaks appeared after annealing over $800\,^{\circ}$ C. Similar results were obtained for STA film on chemically oxidized silicon substrates(not shown in this paper). These crystalline structures of the STA agree with the results in the literatures[5, 6]. The influence of surface treatment of substrates upon the crystallization was investigated in figure 1 (b). For the samples annealed at $900\,^{\circ}$ C for 3min in O_2 , regardless of existence of oxide, diffraction peak patterns were similar aspects.

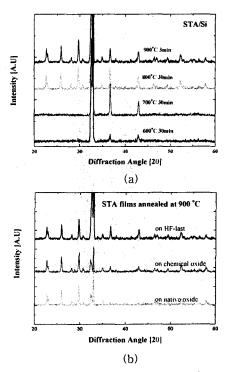


Figure. 1. Phase evolution according to the annealing temperature (a) and surface treatment (b).

Figure 2 shows the AFM images of the surface structure of STA films annealed at different temperatures. RMS variation of the surface roughness were 0.46, 2.8, 0.98 and 0.63nm for the 600, 700, 800, and 900°C annealed samples, respectively. The films have a very smooth surface.

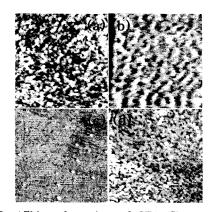


Figure. 2. AFM surface view of STA films on Si as a function of RTA temperature at (a) 600, (b) 700, and (c) $800\,^{\circ}$ C/30min, and (d) $900\,^{\circ}$ C/3min.

Figure 3 shows the results of the high frequency(1 MHz) C-V measurement of STA films on Si at various annealing temperatures. No hysteresis loop was observed for crystallized STA, i.e. annealed over 800°C. The 24nm 900°C/3min films have EOT of 5.7nm and its calculated dielectric constant was 16.5. Large EOT and small relative permitivity might be due to the interfacial SiO₂ and low-ε off-stoichiometric layer formed during annealing process. There was no difference of C-V curves of STA films deposited on oxidized silicon from those of STA on HF-last silicon(not shown in this paper).

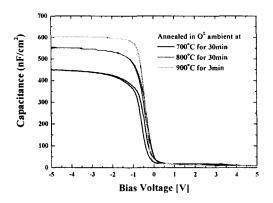


Figure 3. Capacitance-voltage characteristics of STA/Si structure with different annealing temperatures.

The leakage characteristics of crystalline STA films are shown in figure 4. Leakage current of STA thin films directly deposited on Si is around $1.4 \times 10^{-6} \text{A/cm}^2$ at 5V. This relatively high value might be due to polycrystalline structure. But samples which have

structure with chemical oxide show lower leakage current, 3.5×10^{-7} A/cm² at 5V.

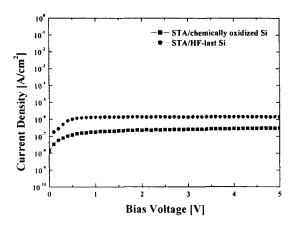


Figure 4. Leakage current densities for STA thin films with respected to the surface treatment. (a) STA deposited on HF-last Si. (b) STA deposited on chemically oxidized Si. Two samples were annealed at $900^{\circ}\text{C}/3\text{min}$ in O_2 .

4. Conclusions

In this study, sol-gel derived $SrTa_2O_6$ thin films were investigated. XRD measurement results indicate that sample annealed below $600\,^{\circ}$ C has amorphous phase and were crystallized over $750\,^{\circ}$ C. C-V curves reveal that EOT and dielectric constant were 5.7nm and 16, respectively, for 24nm thick STA thin film annealed at $900\,^{\circ}$ C. This dielectric constant is lower than previously reported[5], which might be caused by interfacial reaction of STA and Si. Leakage current of STA thin films with chemical oxide is around $3.5 \times 10^{-7} \text{A/cm}^2$ at 5V.

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