

Study on Microstructure of Gd₂O₃ and Nd₂O₃ Compound Film Formed by RF Magnetron Sputtering

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Abstract

In this paper, we report a Gd₂O₃ and Nd₂O₃ compound film formed by RF magnetron sputtering at 500°C. Crystalline structure and morphological features of the film were studied via XRD, TEM, EDS and AFM measurements. By making rare earth oxide compound lattice coincident to silicon, the film deposited were pseudo-single crystal and had (222) preferred orientation on Si (100) substrate.

1. Introduction

As CMOS devices scale down aggressively, much work have been done on finding the replacement of Si-based oxide in gate dielectrics. The rare earth oxides are promising candidates based on thermodynamic energy consideration and a high conduction band offset [1] [2].

Compared with other high-k dielectrics, rare earth oxides are more likely to be crystallized and show a preferred orientation in specific temperature section [3] [6]. In addition to this, lattice parameters of cube rare earth oxides like Gd₂O₃ (10.81Å), Nd₂O₃ (11.08Å), Er₂O₃ (10.55Å) are nearly twice that of silicon (5.43Å). Therefore they are compatible with silicon, and crystalline rare earth oxides can be epitaxially grown on silicon. In this work, by mingling two rare earth oxides Gd₂O₃ and Nd₂O₃, the rare earth oxides compound is lattice coincident to silicon and pseudo-single crystal combined rare earth oxide film is grown on silicon.

2. General Instructions

Experiment procedure

In order to get the lattice parameter of the mixed crystal to match that of silicon, the component of sputtering target should be calculated prudentially. Vegard's law eq.1 was selected to calculate the percentages of Gd₂O₃ and Nd₂O₃ in sputtering target.

$$A=A_1 \cdot X_1 + A_2 \cdot (1-X_1) \quad (1)$$

A is mixed crystal's lattice parameter. A₁ and A₂ are constituent crystal (α , β) lattice parameters respectively. X₁ is the mole percentages of α in mixed crystal. After calculation, target consists of 81% Gd₂O₃ and 19% Nd₂O₃, which meets the requirements. Ideally the lattice parameter of the film sputtering by target is twice that of silicon, which promotes crystallization of film.

The film was deposited in an integrated multi-chamber

ultra high vacuum system using RF magnetron sputtering. The Si (100) substrates were wet chemically cleaned using the RAC method with a final 60s HF (1:10) dip. After that the wafers were immediately loaded into the load-lock and transferred to the UHV growth chamber in order to restrain oxidation of the wafers. The film was deposited at 500°C with deposition pressure of 10×10^{-3} torr in Ar ambient and the sputtering power density was 200W/cm².

Results and discussion

A detailed study of the crystalline structure of the film was conducted by x-ray diffraction (XRD) and transmission electron microscope (TEM) measurements.

The XRD measurements for the as-deposited samples are shown in Fig.1. It presents two intense diffraction peaks at $2\theta=68.70^\circ$ and $2\theta=28.02^\circ$. The main peak at $2\theta=68.70^\circ$ can be associated with reflections for the (004) plane of the silicon substrate, the other peak at $2\theta=28.02^\circ$ exhibits reflections for the (222) plane of the mixed crystal of Gd₂O₃ and Nd₂O₃ [4] [5] [6]. Gd₂O₃ and Nd₂O₃ are cubic crystals, so lattice parameter of mixed crystal can be calculated which is 11.02Å. The lattice parameter of the mixed crystal is almost twice that of the silicon and the relative error is only 1.5%, which indicates that the rare earth oxides compound matches silicon well.

TEM investigations of the sample are shown in Fig.2. The Fig.2 (a) shows the pseudo-single crystal on Si (100) with additional amorphous interfacial layer of around 4nm. The amorphous interface has been improved to be Gd and Nd silicate [4], and can be transformed to crystal by high temperature thermal annealing. Silicon cap layer was deposited on the film before annealing, which can protect the crystal film. Sample after annealing (1000°C/8hours) in nitrogen ambient is shown in Fig.2 (b). The interfacial layer disappeared and rare earth oxide film was crystallized better during annealing step.

In order to identify the accurate composition of the film, we also conducted energy dispersive spectroscopy (EDS). Fig.3 shows each element's component content along the cross of the films. Content of Gd₂O₃ is about four times as much as that of Nd₂O₃, which is expected.

Fig.4 shows the measurement of atomic force microscope (AFM) which characterizes the flatness of film surface. It shows that surface height standard deviation of the layer is only 0.6nm. The layer deposited is flat enough in the follow-up process and can be a seed layer for other film.

3. Conclusions

By combining Gd_2O_3 and Nd_2O_3 , we made rare earth oxide lattice coincident to silicon, and finally got pseudo-single crystal compound film by RF magnetron sputtering in 500°C . XRD and TEM measurements showed that the film was well crystallized and had (222) preferred orientation. We verified chemical component of film by EDS, and AFM showed that the film's surface was smooth enough to deposit other film.

Acknowledgements

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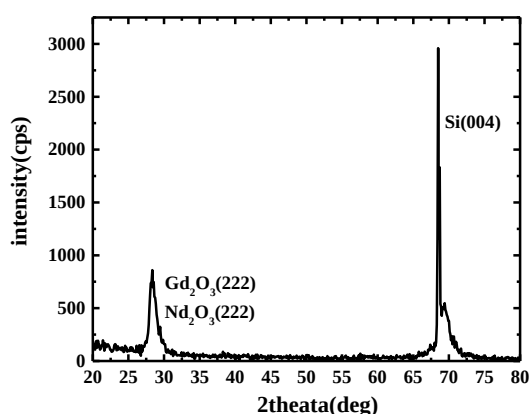


Fig. 1 XRD of rare earth oxide compound film deposited.

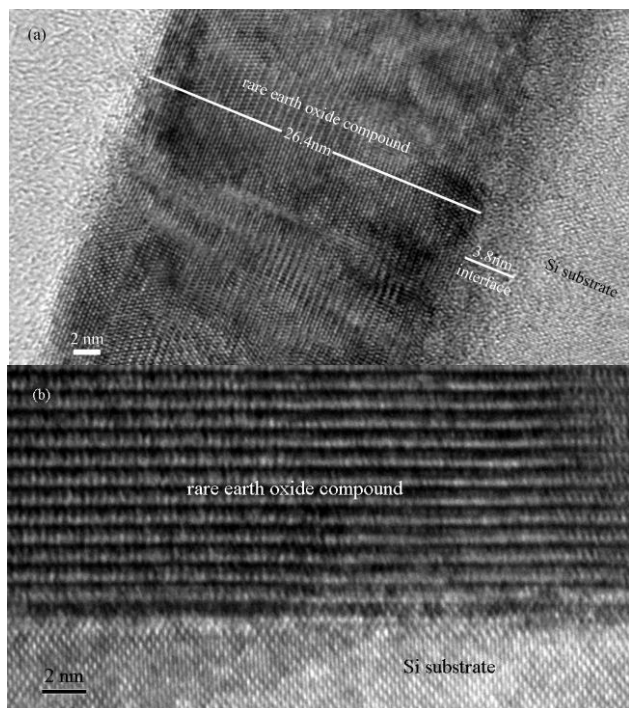


Fig. 2 TEM cross section of rare earth oxide compound film deposited (a) and after annealing ($1000^\circ\text{C}/8\text{hours}$) in nitrogen ambient (b).

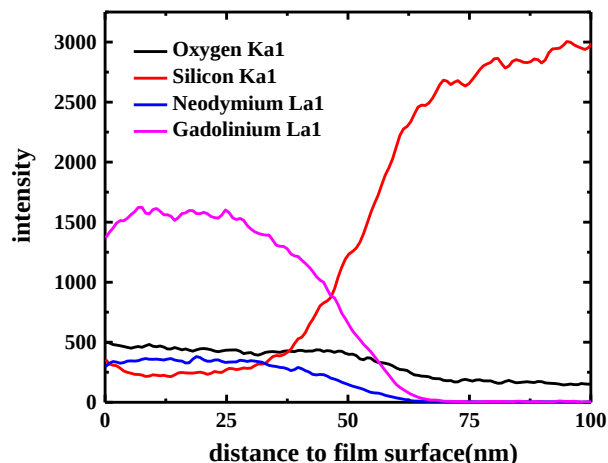


Fig. 3 EDS linescan along the cross of rare earth oxide compound film deposited which is shown in Fig. 2. Scanned area is from surface to Si substrate.

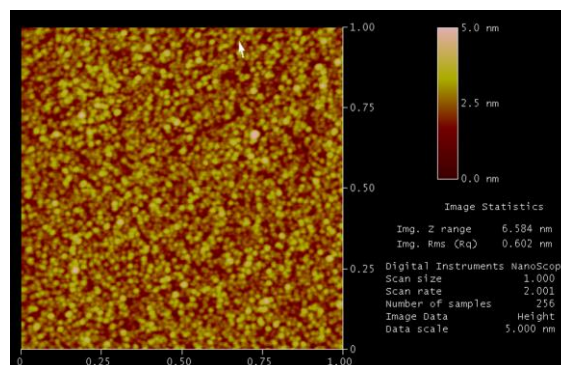


Fig. 4 AFM surface image of rare earth oxide compound film deposited.

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