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Microstructure and electrical properties of Al_2O_3–ZrO_2 composite films for gate dielectric applications

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Abstract

Al_2O_3–ZrO_2 composite films were fabricated on Si by ultrahigh vacuum electron-beam coevaporation. The crystallization temperature, surface morphology, structural characteristics and electrical properties of the annealed films are investigated. Our results indicate that the amorphous and mixed structure is maintained up to an annealing temperature of 900 °C, which is much higher than that of pure ZrO_2 film, and the interfacial oxide layer thickness does not increase after annealing at 900 °C. However, a portion of the Al_2O_3–ZrO_2 film becomes polycrystalline after 1000 °C annealing and interfacial broadening is observed. Possible explanations are given to explain our observations. A dielectric constant of 20.1 is calculated from the 900 °C-annealed ZrO_2–Al_2O_3 film based on high-frequency capacitance–voltage measurements. This dielectric characteristic shows an equivalent oxide thickness (EOT) as low as 1.94 nm. An extremely low leakage current density of ~2×10^{-7} A/cm^2 at a gate voltage of 1 V and low interface state density are also observed in the dielectric film.
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Keywords: ZrO_2–Al_2O_3 film; Electron-beam evaporation; Dielectric properties; MIS structure

1. Introduction

High-κ gate dielectrics substituting for SiO_2 gates in sub-100-nm metal-oxide-silicon field-effect transistors (MOSFETs) have been studied extensively recently because the conventional SiO_2 dielectric faces scaling limits due to excessive gate tunneling current in the coming generations. One of the most promising candidates is ZrO_2 [1–6] due to its high dielectric constant, wide energy band gap [7], and thermodynamic stability in contact with Si [8]. However, pure amorphous ZrO_2 crystallizes at around 500 °C, and so the deposited ZrO_2 layer becomes crystalline after annealing in conventional integrated circuit processes. As a result, grain boundary leakage current and nonuniformity in the κ value as well as film thickness may emerge [9]. More importantly, ZrO_2 is pervious to oxygen. Annealing in an oxygen-rich ambient will lead to fast diffusion of oxygen through the ZrO_2, resulting in interfacial layer growth and consequently, increase in the equivalent oxide thickness (EOT). In comparison, Al_2O_3 films synthesized directly on Si have been reported to remain amorphous up to 1000 °C [10], and Al_2O_3 is known to have a much lower oxygen diffusion coefficient at high temperature [11]. We note that these two dielectric oxides have compensatory characteristics and it is possible to produce a hybrid oxide structure possessing the desirable dielectric properties together with excellent thermal stability as well as high band gap. Several research groups have studied Al_2O_3–ZrO_2 laminated films using atomic layer chemical vapor deposition [12,13], but to our knowledge, the characteristics of Al_2O_3–ZrO_2 composite films fabricated by the simple technique of electron-beam coevaporation have not been reported.

Electron-beam evaporation is an efficient way to evaporate two different materials synchronously to fabricate

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a composite coating. The process allows a temperature in excess of 3500 °C at the source (good for most materials, refractory, dielectric, magnetic, and conductive) and is compatible with ultrahigh vacuum. In the work described here, ultrahigh vacuum electron-beam coevaporation is utilized to synthesize Al_2O_3–ZrO_2 composite films on Si substrate. The crystallization temperature, surface morphology, structural characteristics and electrical properties of the films are studied. Our results demonstrate that the film indeed possesses high crystallization temperature up to 900 °C and excellent electrical properties.

2. Experimental details

P-type Si (100), with resistivity of 10~20 Ω · cm, was used as substrate. It was chemically cleaned by a standard RCA method which consists of two steps. The substrate was cleaned in a NH_4OH:H_2O_2:H_2O (1:1:5) solvent at 70 °C for 10 min, followed by rinsing in deionized (DI) water. It was then cleaned using a HCl:H_2O_2:H_2O (1:1:5) solvent at 80 °C for 10 min, followed by rinsing in DI water again. After this treatment, a 1.2-nm SiO_2 layer (measured by ellipsometry) was formed, which can stabilize and reduce the interfacial states. The substrate was subsequently loaded into the evaporation chamber through a load-lock mechanism. The details of the evaporation apparatus has been described elsewhere [14]. Prior to deposition, the main vacuum chamber UMP500P (made by Balzers) was evacuated to below 10^{-7} Pa. High-purity sintered ZrO_2 and Al_2O_3 pellets were coevaporated simultaneously by two electron guns in high vacuum and deposited synchronously onto the substrate at room temperature. The deposition rate of ZrO_2 and Al_2O_3 measured by the quartz crystal monitor was about 0.06 and 0.03 nm/s, respectively, and the duration was 100 s. After deposition, rapid thermal annealing (RTA) in a N_2 ambient was performed for 2 min at different temperatures ranging from 800 to 1100 °C. X-ray diffraction (XRD) was conducted to determine the crystallization temperature on a Siemens 500/501 diffractometer with a Cu K X-ray at a fixed incident angle of 1° and atomic force microscopy (AFM) was used to characterize the surface morphology. An AFM (SPA400) was operated in the contact mode at room temperature under dry nitrogen. High-resolution transmission electron microscopy (HRTEM, Philips CM200FEG) was used to study the interfacial quality and the morphology of the annealed Al_2O_3–ZrO_2 films. The electrical properties were evaluated using an HP 4284A inductance–capacitance–resistance meter and HP 4156B semiconductor parameter analyzer.

3. Results and discussion

Fig. 1 displays the XRD pattern obtained from samples annealed at different temperatures. Up to an annealing temperature of 900 °C, no diffraction peaks can be observed, indicating that the amorphous structure is maintained in the Al_2O_3–ZrO_2 films. At 1000 °C, some relatively broad and weak multiple diffraction peaks emerge at 30° and 34°, which can be attributed to the ZrO_2 (101), (002) and (110) planes. The large width and low intensity suggest that very small grains are locally embedded in the amorphous structure. With increasing annealing temperature, the intensities of the ZrO_2 peaks increase and some other peaks appear at 50° attributable to ZrO_2 (112) and (200). When the temperature reaches 1100 °C the main peak becomes sharp and more intense, implying an increase of the volume fraction of the crystalline phase in the Al_2O_3–ZrO_2 structure. From the position of the peak, it can be inferred that the crystalline phase is tetragonal ZrO_2, but not cubic ZrO_2 or Al_2O_3. Our previous work has shown that the pure ZrO_2 films fabricated by electron-beam evaporation begin to crystallize at 700 °C [15]. It is obvious that the crystallization onset temperature of ZrO_2 film increases substantively when Al_2O_3 is incorporated. These results clearly suggest that crystallization of the Al_2O_3–ZrO_2 composite structure during postannealing is effectively suppressed. It is due to the fact that addition of Al_2O_3 into ZrO_2 matrix affects nucleation and growth of ZrO_2 grains from the composite matrix, so mixing of Al_2O_3 with ZrO_2 will raise the crystallization onset temperature.

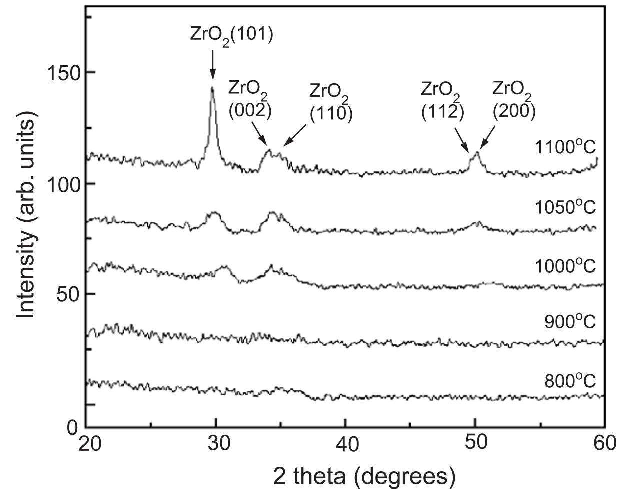

Fig. 1. XRD pattern of the samples after RTA at different temperatures.

It is known that crystallization of an oxide causes an increase in both leakage current and surface roughness, and so atomic force microscopy (AFM) was used to examine the surface morphology of the samples annealed at different temperatures. Fig. 2 depicts the 1×1 μm AFM images of the same set of samples analyzed by XRD. In Fig. 2(a), the 900 °C-annealed sample shows no obvious crystallization. In contrast, the 1000 °C-annealed sample shown in Fig. 2(b) is coarser than the former and exhibits an apparent partial crystalline structure. Subsequently, a larger area of 50×50 μm was scanned, and the surfaces of all the samples are

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