Effects of O_2/Ar ratio and annealing temperature on electrical properties of Ta_2O_5 film prepared by magnetron sputtering*

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(Received 12 July 2012; revised manuscript received 5 August 2012)

The effects of the oxygen–argon ratio on electric properties of Ta_2O_5 film prepared by radio-frequency magnetron sputtering were investigated. The Ta_2O_5 partially transforms from the amorphous phase into the crystal phase when annealing temperatures are 800 °C or higher. The lattice constant of Ta_2O_5 decreases with the increase of the O_2/Ar ratio, which indicates that oxygen gas in the working gas mixture contributes to reducing the density of oxygen vacancies during the deposition process. For the films deposited in working gas mixtures with different O_2/Ar ratios and subsequently annealed at 700 °C, the effective dielectric constant is increased from 14.7 to 18.4 with the increase of the O_2/Ar ratio from 0 to 1. Considering the presence of an SiO_2 layer between the film and the silicon substrate, the optimal dielectric constant of Ta_2O_5 film was estimated to be 31. Oxygen gas in the working gas mixture contributes to reducing the density of oxygen vacancies, and the oxygen vacancy density and leakage current of Ta_2O_5 film both decrease with the increase of the O_2/Ar ratio. The leakage current decreases after annealing treatment and it is minimized at 700 °C. However, when the annealing temperature is 800 °C or higher, it increases slightly, which results from the partially crystallized Ta_2O_5 layer containing defects such as grain boundaries and vacancies.

Keywords: Ta_2O_5 film, magnetron sputtering, *C*–*V*, oxide charge

PACS: 77.55.+f, 84.37.+q, 68.35.D **DOI:** 10.1088/1674-1056/22/2/027701

1. Introduction

Due to its high dielectric constant, chemical stability, high breakdown fields, and low leakage current, tantalum pentoxide (Ta_2O_5) is the strongest candidate among the high dielectric constant (high-*k*) materials. The electrical performance of the Ta_2O_5 films is greatly influenced by oxide charges in the bulk of Ta_2O_5 as well as by the interface state charges. Oxygen vacancies are the principal source of oxide charges and bulk defects in Ta_2O_5, and they can be efficiently reduced in an oxidizing ambience at the beginning or during the deposition step and by post-annealing treatments. Therefore, the current–voltage (*I*–*V*) characteristics of Ta_2O_5 film are improved after oxygen annealing. The effect of oxygen annealing also depends on the annealing temperature, and annealing leads to a very pronounced decrease of the leakage current of Ta_2O_5 films at room temperature.[1,2] However, an SiO_2 interfacial layer will form between the high-*k* Ta_2O_5 layer and the Si substrate during oxygen annealing, which results in decreased capacitance due to its low dielectric constant.[3,4] This interfacial layer has a detrimental effect on the capacitance of devices and is a prime impediment in the use of high-*k* materials.

Researchers have prepared Ta_2O_5 films by various methods, such as sputtering,[5] thermal oxidation,[6] low-pressure chemical vapor deposition (LPCVD),[7] anodization,[8] and atomic layer deposition (ALD).[9] Although uniform, dense Ta_2O_5 film can be obtained by the thermal oxidation of tantalum in oxygen ambience, the thermal oxidation method has some shortcomings, such as high process temperature, slow oxidation rate, and relatively long temperature ramping time. Anodic oxidation can be obtained either in an aqueous or non-aqueous solution. The oxide is grown by applying a voltage between the substrate, used as the anode electrode, and a cathode immersed in an electrolyte. Anodic oxidation with high deposition rate and low temperature can lead to a significant density of defect traps in the oxide. An LPCVD reaction occurs between two gaseous species on the substrate surface at low pressure and low deposition temperature. However, low deposition temperatures lead to low quality films, containing reaction by-products (mainly hydrogen) and dangling bonds, and having non-stoichiometric composition. ALD is based on chemical reactions at the surface of a substrate during which the reactants are alternatively transported as vapor pulses.[10] The desired solid film is formed via saturating, self-controlled surface reactions allowing a homogeneous distribution of the active species throughout the surface area. Therefore, the ALD technique allows the controlled growth of solid films with excellent thickness uniformity, the possibility of conforming to irregular surfaces, and a low defect density. Nevertheless, the film deposition rate is low and the film preparation cost is high.

Tantalum pentoxide thin films can be obtained by sputtering from a tantalum or tantalum oxide target at low pressure in the presence of an oxygen atmosphere with or without an

*Project supported by the National Natural Science Foundation of China (Grant No. 61076055), the Program for Innovative Research Teams in Zhejiang Normal University, and the Open Project Program of Surface Physics Laboratory (National Key Laboratory) of Fudan University (Grant No. FDS KL2011_04).

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inert gas (such as argon). This method is based on cathodic erosion by bombardment with ionized gas molecules from a discharge.[11,12] In general, Ta_2O_5 from sputtering deposition on a silicon substrate has been achieved in two ways. One is direct sputtering when the substance to be sputtered is tantalum pentoxide, and the other is the reactive sputtering of a Ta target in a mixture of Ar and O_2 gas.[13] The deposition rate depends mainly on the radio-frequency (RF) power density and on the gas pressure. This method has the advantage that it can be performed at relatively low temperatures (150 °C∼400 °C). Among the above mentioned deposition techniques, the standard magnetron sputtering has high resource usability, high deposition velocity, large area deposition capability, and a safe deposition process. The deposition rate of Ta_2O_5 film can be significantly improved using a Ta_2O_5 target in an RF magnetron sputtering process. But some problems may be induced by this technique: the film's stoichiometry is not easy to control, and the film generally has a low density.

The purpose of this paper is to investigate the effect of the ratio of oxygen and argon on the electrical properties of Ta_2O_5 film prepared by RF sputtering using a Ta_2O_5 target. Special attention is focused on the crystallization effects under different annealing temperatures and their impact on the capacitance–voltage (C–V) characteristics and leakage current. The close relationship between the electric and dielectric properties of Ta_2O_5 film and the film's oxygen vacancies are also discussed.

2. Experimental details

Si (100) wafer (p-type) substrates with a resistivity of about 5 Ω·cm and area of about 25 mm×25 mm were first degreased in detergent. The substrates were processed by standard RCA cleaning technology for a silicon wafer. After rinsing by deionized water and drying by nitrogen gas, they were inserted immediately into the pretreatment chamber. In order to eliminate the oxide and water vapor absorbed on the surface of the Si substrate, it was anti-sputtered in the pretreatment chamber and then transported to the sputtering chamber by means of a magnetically coupled transfer rod. The apparatus used is an RF (13.56 MHz) magnetron sputtering system. The distance between the target and the substrate is about 7.5 cm. The targets used here are high-purity Ta_2O_5 (99.99%) and metal aluminum (Al, 99.9%). The sputtering chamber and the pretreatment chamber were evacuated first down to 5.0×10^{-5} Pa. The sputter cleaning of the substrate and target surfaces was carried out prior to the film deposition. After surface cleaning, Al with a thickness of about 500 nm is sputtered onto the whole back surface of the Si wafer. Then, a low resistivity Ohmic contact is added, followed by a temperature treatment at 500 °C for 5 min in Ar atmosphere. In order to eliminate the oxide and water vapor absorbed on the surface of the targets, they were sputtered in the sputtering chamber for 10 min before the growth of Ta_2O_5 films. The substrate temperature was maintained at room temperature (RT) and the working gas is the mixed gas (Ar and O_2). The O_2/Ar ratio was adjusted between 0 and 1. The working gas pressure was fixed at 0.3 Pa and the sputtering power density was set to 2.5 W/cm^2. The film's thickness can be accurately controlled by optimizing the sputtering parameters, and the typical thickness is 18 nm. After deposition, *in-situ* thermal annealing treatments were applied for 30 min in the pretreatment chamber under a pure argon atmosphere between 600 °C and 800 °C. The Schottky contacts were formed on the front surface by sputtering Al with a thickness of 500 nm as dots with a diameter of about 1.0 mm through a metal shadow mask.

C–V measurements were performed using an Agilent 4284A precision LCR meter at RT and a small sinusoidal test signal of 50 mV. I–V measurements were performed using a Keithley 2601 Sourcemeter at RT. C–V and I–V measurements were carried out with the help of a microcomputer through a GPIB interface converter. The X-ray diffraction (XRD) data were obtained using a diffractometer (X'Pert PW 3040/60, Philips). The thickness of the film was measured by a surface profiler (Alpha-Step IQ), and the morphology of the film was analyzed by atomic force microscopy (AFM) (NT-MDT).

3. Results and discussion

3.1. Structure and morphology

The crystalline properties of the samples were investigated by XRD. Glancing incidence XRD was chosen to maximize the crystalline volume intersected by the X-ray beam. The sample was fixed at an angle (∼0.26°) close to the critical angle, and 2θ scans were performed. The XRD patterns of the samples deposited in pure Ar gas ambience and subsequently annealed at 600 °C–800 °C are shown in Fig. 1. An obvious influence of annealing temperature on crystalline structure can be observed. For the sample deposited at RT and annealed below 700 °C, only a broad diffraction peak at 28.5° was observed, which indicates the absence of crystalline phases. For the sample annealed at 800 °C, the XRD spectra present several typical Ta_2O_5 Bragg peaks (β-Ta_2O_5) corresponding to (001), (200), (201), and (202), whose 2θ values are located around 22.84°, 28.56°, 36.82°, and 55.73°, respectively.[14] These results demonstrate that the Ta_2O_5 is transformed from the amorphous phase into the crystal phase when the annealing temperature is 800 °C or higher.

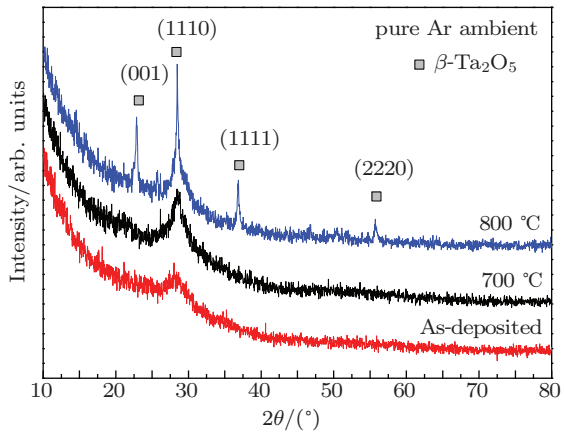


Fig. 1. (color online) XRD spectra of the sample deposited in pure Ar ambience and subsequently annealed at 600 °C–800 °C.

When the working gas of pure argon is replaced by the mixed gas of oxygen and argon, the effect of the annealing temperature on the crystallization of Ta_2O_5 is not changed. When the O_2/Ar ratio increases from 0 to 1.0, the diffraction peak of crystalline plane (200) for the sample annealed at 800 °C shifts slightly from 28.56° to 28.64°, and the corresponding lattice constant (a_0) decreases from 0.626 nm to 0.622 nm by the Bragg formula. As shown in Table 1, the value of a_0 decreases with the increase of O_2/Ar ratio. It is especially noteworthy that the value of a_0 does not change when the O_2/Ar ratio is larger than 0.8. A large lattice constant implies that there are many defects, such as oxygen vacancies. When the O_2/Ar ratio is smaller than 0.5, there are many oxygen vacancies in Ta_2O_5 film. However, the density of oxygen vacancies in Ta_2O_5 film decreases rapidly when the O_2/Ar ratio is larger than 0.8. These results indicate that oxygen gas in the working gas mixture contributes to reducing the density of oxygen vacancies during the deposition process.

Table 1. Diffraction peak and lattice constant of crystalline plane (200) for the sample annealed at 800 °C in working gas mixtures with different O_2/Ar ratios.

O_2/Ar ratio	0	0.3	0.6	0.8	1.0
Diffraction peak ($2\theta_{200}$)/(°)	28.56	28.61	28.64	28.65	28.65
Lattice constant (a_0)/nm	0.626	0.624	0.623	0.622	0.622

The microstructure evolution of Ta–Ta_2O_5 alloys during arc melting and the following annealing process for different oxygen contents has been well analyzed by Jehn *et al.*[15] In order to investigate the effects of oxygen content on the surface properties of Ta_2O_5 thin films, AFM was performed. The AFM scan area was 2 μm×2 μm. Figures 2(a)–2(d) show AFM images of samples deposited in working gas with different O_2/Ar ratios and subsequently annealed at 700 °C in pure Ar ambience. For the samples deposited in pure Ar gas ambience, i.e. O_2/Ar=0, the surface morphology is rough, as shown in Fig. 2(a), with many cracks and defects. With the increase of the O_2/Ar ratio, the average roughness is decreased. At lower O_2/Ar ratios, fewer oxygen atoms diffuse to oxygen vacancies in Ta_2O_5 film, leading to greater roughness. At higher O_2/Ar ratio, more oxygen atoms diffuse to oxygen vacancies and fill them, which makes the roughness of the thin films decrease. This decrease in roughness with increasing O_2/Ar ratio is consistent with the XRD studies.

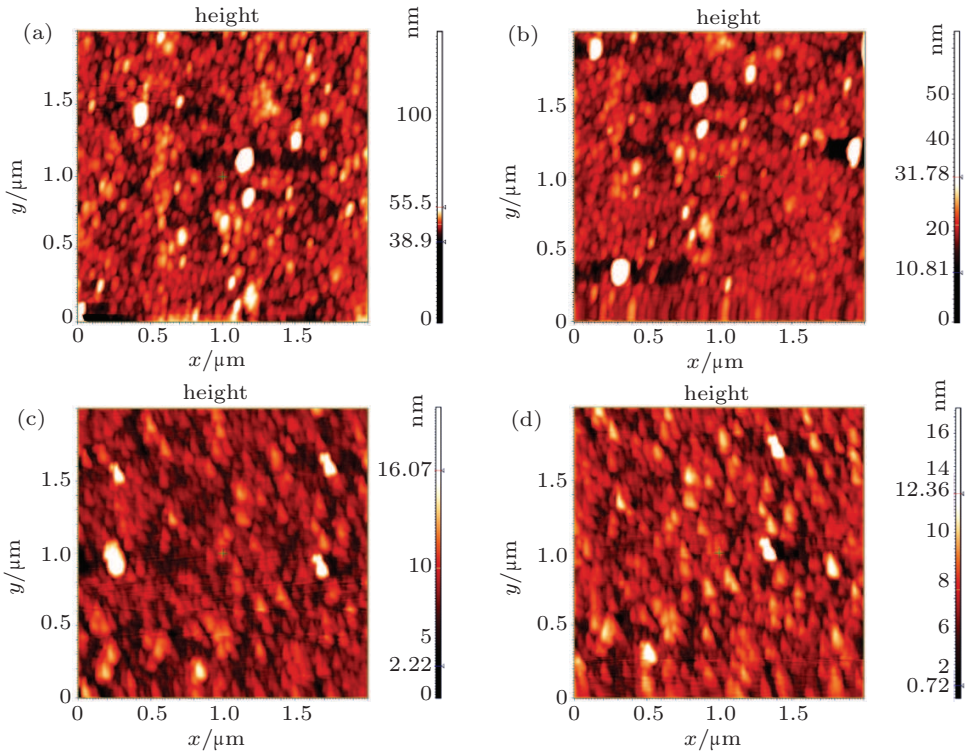


Fig. 2. (color online) AFM images of the sample deposited in working gas mixtures with different O_2/Ar ratios and subsequently annealed at 700 °C in pure Ar ambience: (a) 0; (b) 0.3; (c) 0.5; (d) 0.8.

3.2. *C–V* and dielectric characteristics

The electrical properties of the Ta_2O_5 dielectric films were evaluated as functions of the O_2/Ar ratio and annealing temperature through *C–V* and *I–V* analyses. Figure 3 shows *C–V* curves measured at 1-MHz frequency for an Al/Ta_2O_5/p-Si MOS capacitor structure. The Ta_2O_5 film was deposited in pure Ar ambience, and subsequently annealed at 600 °C–800 °C. The applied voltage was swept from +2 V to −5 V and then was swept back to +2 V. The accumulation capacitance of the annealed sample is larger than that of the as-deposited sample due to the increased amount of Ta-silicate, which has a high dielectric constant. In general, the MOS capacitance is affected by interface states of the interfacial layer between the Ta_2O_5 and the Si substrate. The enhanced accumulation capacitance is due to the increased concentration of silicate with its high dielectric constant.[16]

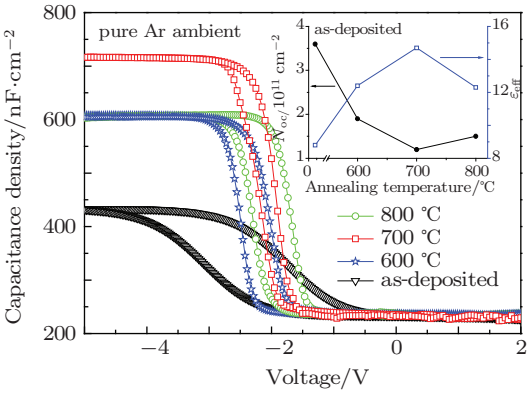


Fig. 3. (color online) *C–V* characteristics at 1-MHz measurement frequency for the sample deposited in pure Ar ambience and subsequently annealed at 600 °C–800 °C. The inset shows the oxide charge density (N_{oc}) and the effective dielectric constant (ε_{eff}) resulting from different annealing temperatures.

Oxygen vacancies are the principal source of oxide charges and bulk defects in Ta_2O_5, and they can be efficiently reduced in an oxidizing ambience at the beginning or during the deposition step and by post-annealing treatments. Figure 4 shows the *C–V* characteristics of the samples deposited in working gas with different O_2/Ar ratios and subsequently annealed at 700 °C in pure Ar ambience. With an increase of the O_2/Ar ratio, the *C–V* hysteresis width (ΔV_{FB}) decreases, and the accumulation capacitance C_{acc} increases.

The annealing temperature has a significant effect on the charge trapping of high-*k* material. The most popular method to obtain the oxide charge density N_{oc} is to deduce it from the voltage shift of a *C–V* curve. The oxide charge density can be quantitatively calculated from the *C–V* curve using the following formula [17]

$$N_{oc} = C_{acc}\Delta V_{FB}/qA, \tag{1}$$

where C_{acc} is the accumulation capacitance per unit area, ΔV_{FB} is the *C–V* hysteresis width, q is the electron charge, and A is the effective electrode area. The inset in Fig. 3 shows the oxide charge density of MOS stacks. The oxide charge density is 3.6×10^{11} cm^{-2} for the as-deposited sample. N_{oc} decreases after annealing treatment and is minimized (1.2×10^{11} cm^{-2}) at the annealing temperature of 700 °C. After annealing in pure Ar ambience, N_{oc} is significantly reduced, which is due to two possibilities: a net annealing (real reduction of N_{oc} as a result of an improvement of the oxide parameters) or a partial charge compensation during the annealing. When annealing temperature is 800 °C or higher, N_{oc} slightly increases to 1.5×10^{11} cm^{-2}. These results indicate that there might be a critical temperature for annealing treatment. The charge trapping may be attributed to the structural defects in the as-deposited films. However, the charge trapping density for the sample annealed at 800 °C increases slightly because the partially crystallized Ta_2O_5 layer contains defects such as grain boundaries and vacancies. The as-deposited Ta_2O_5 is amorphous, but annealing at temperatures 800 °C or higher causes pronounced crystallization. Nevertheless, polycrystalline gate dielectrics are not preferable as producing samples with similar grain statistics is unachievable. Annealing at 800 °C or above reduces the interface state density but at the same time introduces many oxide traps due to crystallization effects.

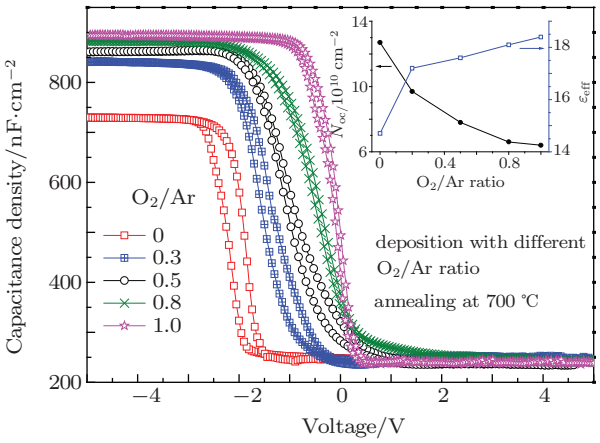


Fig. 4. (color online) *C–V* characteristics for the sample deposited in working gas mixtures with different O_2/Ar ratios and subsequently annealed at 700 °C in pure Ar ambience. The inset shows the oxide charge density (N_{oc}) and the effective dielectric constant (ε_{eff}) resulting from different O_2/Ar ratios.

As shown in the inset of Fig. 4, the oxide charge density N_{oc} decreases with the increase of the O_2/Ar ratio. When the O_2/Ar ratio increases from 0 to 0.8, N_{oc} decreases from 1.27×10^{11} cm^2 to 6.4×10^{10} cm^2. When the O_2/Ar ratio is larger than 0.8, the reduction of N_{oc} is very slow. Generally speaking, the electrically active defects in Ta_2O_5 are oxygen vacancies and the oxidizing nature of oxygen treatment due to the decomposition of Ta_2O_5 in the sputtering process. In the oxygen ambience during the film deposition process, the oxygen diffuses through Ta_2O_5 and reduces the density of oxygen

vacancies observed as the oxide charges. When the O_2/Ar ratio of the mixture of working gas is lower than 0.8, the oxygen vacancy density rapidly decreases with increasing O_2/Ar ratio.

The effective dielectric constant ($\varepsilon_{\rm eff}$) of the RF sputtered Ta_2O_5 film is strongly dependent on the fabrication process and post-deposition annealing treatment. As shown in the inset of Fig. 3, $\varepsilon_{\rm eff}$ is 8.8 for as-deposited film. With the increase of annealing temperature, $\varepsilon_{\rm eff}$ increases to its maximum value (14.7) at 700 °C annealing, and then begins to reduce to 12.3 at 800 °C annealing. For the films deposited in working gas with different O_2/Ar ratios and subsequently annealed at 700 °C, $\varepsilon_{\rm eff}$ is increased from 14.7 to 18.4 with the increase of O_2/Ar ratio from 0 to 1. The dielectric constant of Ta_2O_5 thin film has been reported in the range (14–60).[18–20] The dielectric properties of Ta_2O_5 thin film strongly depend on the fabrication method, post-deposition annealing treatment, crystallization, film-substrate interface characteristics, etc. A poor interface between dielectric film and silicon has a great many defect-related trapped charges. In the present case, the Ta_2O_5 thin films were deposited on p-Si substrate to analyze the effects of Ta_2O_5 film-silicon interface on the dielectric properties. The films annealed below 700 °C show a higher dielectric constant than do the as-deposited films because of the decreased oxygen vacancy density. However, the films annealed at 800 °C show a low dielectric constant, which arises from the partially crystallized film's defects such as grain boundaries and vacancies. Similarly, the increase of the dielectric constant for the films deposited in O_2/Ar mixture of working gas is due to the decreased oxygen vacancy density. When the film is deposited in oxygen ambience, the oxygen diffuses through the Ta_2O_5 and reduces the oxygen vacancies, which are observed as a fixed oxide charge.

The dielectric constant obtained here is smaller than that reported in references [18]–[20], which may be due to the formation of a uniform oxide layer between the film and silicon substrate.[21,22] For a planar capacitor, the charge storage capacity is defined by

$$C = \varepsilon_0 \varepsilon_r A/d, \tag{2}$$

where ε_0 is vacuum permittivity, $\varepsilon_{\rm r}$ is relative permittivity, and d is thickness. Considering the presence of the SiO_2 layer, the measured capacitance ($C_{\rm eff}$) of MOS capacitor should be a series combination of Ta_2O_5 film capacitance ($C_{Ta_2O_5}$) and SiO_2 layer capacitance (C_{SiO_2}), i.e.,

$$1/C_{\rm eff} = 1/C_{Ta_2O_5} + 1/C_{SiO_2}. \tag{3}$$

Assuming that the SiO_2 layer is about 2-nm thick,[21,22] the intrinsic dielectric constant of Ta_2O_5 film is deduced to be about 11 and 31, respectively, for the unannealed film deposited in pure Ar ambience and the film deposited in working gas with a 1:1 O_2/Ar ratio and subsequently annealed at 700 °C.

3.3. Leakage current characteristics

The leakage current of the as-deposited Ta_2O_5 layer in an Al-Ta_2O_5-Si capacitor is usually large and a post-annealing treatment is needed to reduce this current. The leakage current is one of the most important parameters of Ta_2O_5 based capacitors, and it is very sensitive to material imperfections such as oxygen vacancies, which increase the current leakage. After post-annealing treatment, the leakage current significantly decreases, as shown in Fig. 5. For the sample annealed at 700 °C, the leakage current density is minimized (8.8×10^{-9} A/cm^2 at the gate voltage of −4 V). However, the leakage current for the sample annealed at 800 °C is a little higher, which results from partial crystallization of the Ta_2O_5 layer. Similarly, the decreased leakage current of the film deposited in O_2/Ar working gas mixture is due to the decreased oxygen vacancy density. When the O_2/Ar ratio increases from 0 to 0.8, the leakage current lessens from 8.5×10^{-9} A/cm^2 to 2.5×10^{-9} A/cm^2 at the gate voltage of −4 V, as shown in Fig. 6. However, the oxygen vacancy density and leakage current both decrease very slowly after the O_2/Ar ratio gets larger than 0.8.

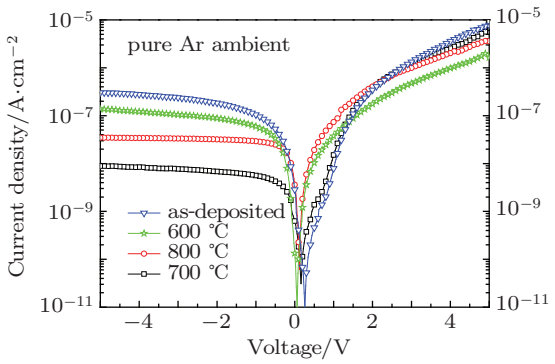


Fig. 5. (color online) *I–V* characteristics of the sample deposited in pure Ar ambience and subsequently annealed at 600 °C–800 °C.

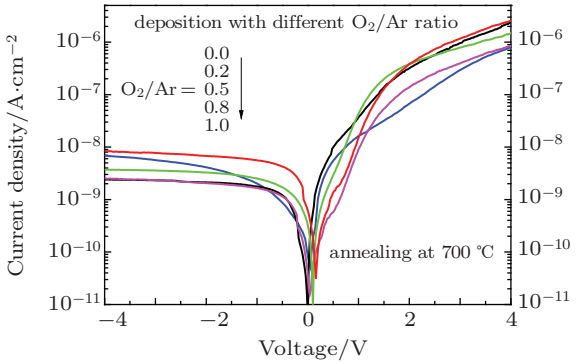


Fig. 6. (color online) *I–V* characteristics of the samples deposited in working gas mixtures with different O_2/Ar ratios and subsequently annealed at 700 °C in pure Ar ambience.

In general, the leakage current of thin film depends strongly on material-related parameters such as composition, structure, and imperfections. Reduced defect density was found to depend on annealing temperature, thus the decreased

leakage current of Ta_2O_5 thin film annealed at temperatures lower than 700 °C can be attributed to decreased defect density. The films annealed at temperatures lower than 800 °C exhibit higher leakage current, which arises from the grain boundaries of partially crystallized thin film. These grain boundaries serve as a new leakage current path, resulting in poor leakage current characteristics.

The tantalum to oxygen ratio in the as-deposited film is close to chemically stoichiometric Ta_2O_5, as oxygen sputtering partial pressure is large enough. These results indicate that oxygen vacancies and leakage current of Ta_2O_5 film can be significantly reduced at large oxygen sputtering partial pressure. Film sputtering at high oxygen partial pressure might be closely related to the decrease in the frequency of electron traps (most likely oxygen vacancies and/or broken bonds) and to the improvement of the film microstructure due to increased film density. The leakage current reduction at high oxygen partial pressure may be attributed to the removal of certain structural defects present in Ta_2O_5. Therefore, it is reasonable to assume that sputtering at high oxygen partial pressure may well affect the concentration of the oxygen vacancies and imperfect bonds, and consequently reduce leakage current.

4. Conclusions

The effects of O_2/Ar ratio and annealing temperature on electrical properties of Ta_2O_5 film prepared by RF magnetron sputtering using Ta_2O_5 target were investigated. The XRD results show that Ta_2O_5 partially transforms from the amorphous phase into the crystal phase when the annealing temperature is 800 °C or higher. The lattice constant of Ta_2O_5 decreases with the increase of O_2/Ar ratio, which indicates that oxygen gas in the working gas mixture contributes to reducing the density of oxygen vacancies during the deposition process of Ta_2O_5 film. With the increase of O_2/Ar ratio, the average roughness is decreased. At lower O_2/Ar ratio, the less oxygen atoms diffuse to oxygen vacancies in Ta_2O_5 film, leading to larger roughness. At higher O_2/Ar ratio, a sufficient number of oxygen atoms can diffuse to oxygen vacancies and fill them, which makes the roughness of the thin films decrease.

The effective dielectric constant of the RF sputtered Ta_2O_5 film is strongly dependent on the fabrication process and post-deposition annealing treatment. For the film deposited in the working gas mixture with different O_2/Ar ratio and subsequently annealed at 700 °C, the effective dielectric constant is increased from 14.7 to 18.4 with the increase of O_2/Ar ratio from 0 to 1. Considering the presence of the SiO_2 layer between the film and silicon substrate, the optimal dielectric constant of Ta_2O_5 thin film was estimated to be 31. The increase of dielectric constant for the film deposited in O_2/Ar working gas mixture is due to the decrease of oxygen vacancy density. When the film was deposited in ambient oxygen, the oxygen diffused through Ta_2O_5 and reduced the oxygen vacancies, observed as fixed oxide charge.

The oxygen vacancy density decreases after annealing treatments, which resulted in the decrease of the leakage current. The oxygen vacancy density and leakage current decrease after annealing treatment and their magnitudes achieve minimum values at the annealing temperature of 700 °C. However, when the annealing temperature is 800 °C or higher, they increase slightly, which results from the partially crystallized Ta_2O_5 layer containing the defects such as grain boundaries and vacancies. The film sputtering at high oxygen partial pressure might be closely related to the decrease of the electron traps (most likely oxygen vacancies and/or broken bonds) and to the improvement of the film microstructure due to film densification. The leakage current reduction at high oxygen partial pressure may be attributed to the removal of certain structural defects present in Ta_2O_5.

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