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INFLUENCE OF ZIRCONIUM DOPING ON THE DIELECTRIC PROPERTIES OF HAFNIUM OXIDE FILMS

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Abstract. A comparison of the dielectric characteristics (relative permittivity, dielectric loss tangent, band gap, leakage current and breakdown voltage) of hafnium and hafnium-zirconium oxide films was carried out. It is shown that pulsed reactive magnetron sputtering of a Hf target in an Ar/O_2 working gas environment can be used to obtain HfO_x films with a relative permittivity of ε = 12.5–16.0 and ε = 12.0–14.0 at frequencies of F = 1 kHz and F = 1 MHz, respectively, with a dielectric loss tangent of tgα = 0.012–0.022 (F = 1 kHz) and tgα = 0.053–0.062 (F = 1 MHz), a leakage current density of $J_L = (1.0–3.0) \cdot 10^{-3}$ A/m^2 at an electric field strength of $E = 5 \cdot 10^7$ V/m, with a band gap of E_g = 5.85–5.87 eV and a breakdown field strength of $E_{br} = (2.1–2.4) \cdot 10^8$ V/m. Doping of hafnium oxide with zirconium (40 at.%) made it possible to reduce the dielectric loss tangent to 0.008–0.012 (F = 1 kHz) and to 0.04–0.05 (F = 1 MHz), the leakage current density to $(3–5) \cdot 10^{-5}$ A/m^2, and increase the breakdown voltage to $(2.5–3.0) \cdot 10^8$ V/m. At the same time, a slight increase in the relative permittivity of the films to 14–16 was observed at frequencies of 1 kHz and 1 MHz due to a decrease in frequency dispersion from 1.15 to values less than 1.10 and an increase in E_g to 5.86–5.89 eV.

Keywords: thin film, hafnium oxide, hafnium zirconium oxide, reactive magnetron sputtering, composite target, dielectric properties.

Conflict of interests. The authors declare no conflict of interests.

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ВЛИЯНИЕ ЛЕГИРОВАНИЯ ЦИРКОНИЕМ НА ДИЭЛЕКТРИЧЕСКИЕ СВОЙСТВА ПЛЕНОК ОКСИДА ГАФНИЯ

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Аннотация. Проведено сравнение диэлектрических характеристик (относительной диэлектрической проницаемости, тангенса угла диэлектрических потерь, ширины запрещенной зоны, тока утечки и пробивного напряжения) пленок оксидов гафния и гафния-циркония. Показано, что импульсным реактивным магнетронным распылением Hf-мишени в среде рабочих газов Ar/O_2 могут быть получены пленки HfO_x с относительной диэлектрической проницаемостью ε = 12,5–16,0 и ε = 12,0–14,0 на частотах F = 1 кГц и F = 1 МГц соответственно, с тангенсом угла диэлектрических потерь tgφ = 0,012–0,022 (F = 1 кГц) и tgφ = 0,053–0,062 (F = 1 МГц), плотностью тока утечки $J_L = (1,0–3,0) \cdot 10^{-3}$ А/м^2 при напряженности электрического поля $E = 5 \cdot 10^7$ В/м, с шириной запрещенной зоны E_g = 5,85–5,87 эВ и напряженностью поля пробоя $E_{пр} = (2,1–2,4) \cdot 10^8$ В/м. Легирование оксида гафния цирконием (40 ат.%) позволило уменьшить тангенс угла диэлектрических потерь до 0,008–0,012 (F = 1 кГц) и до 0,04–0,05 (F = 1 МГц), плотность тока утечки – до $(3–5) \cdot 10^{-5}$ А/м^2, увеличить пробивное напряжение до $(2,5–3,0) \cdot 10^8$ В/м. При этом наб-

людалось незначительное повышение относительной диэлектрической проницаемости пленок до 14–16 на частотах 1 кГц и 1 МГц за счет уменьшения частотной дисперсии от 1,15 до значений менее 1,10 и увеличения E_g до 5,86–5,89 эВ.

Ключевые слова: тонкая пленка, оксид гафния, оксид гафния-циркония, реактивное магнетронное распыление, составная мишень, диэлектрические свойства.

Конфликт интересов. Авторы заявляют об отсутствии конфликта интересов.

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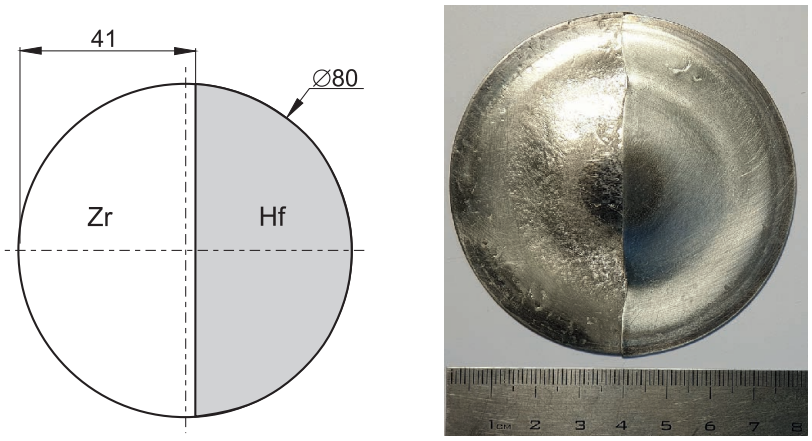
Introduction

In recent years, there has been an increase in research aimed at studying the properties and developing technologies for depositing thin films of dielectrics with high permittivity (*high-k* dielectrics), and, in particular, hafnium oxide (HfO_2) [1]. Hafnium oxide has high permittivity ($\varepsilon \approx 16–25$), a large band gap ($E_g$ = 5.6–5.8 eV), high thermodynamic stability at the interface with silicon, and high energy barriers for electrons and holes relative to silicon (2.0 and 2.5 eV, respectively) [1–3]. The combination of these properties creates great prospects for the use of amorphous HfO_2 films in silicon microelectronics as an alternative to silicon dioxide in metal-oxide-semiconductor (MOS) structures. As a result, the electrical properties of HfO_2 thin films have been extensively studied, and it has been shown that amorphous hafnium oxide has improved dielectric properties compared to the strong crystalline phase [4]. However, amorphous hafnium oxide does not have sufficient thermal stability and tends to crystallize at temperatures of 400–450 °C [5, 6]. Crystalline HfO_2 is polymorphic and can have three modifications of the crystal lattice in the bulk state: monoclinic, tetragonal and cubic [7]. In this case, the dielectric characteristics depend on the forming structure of the deposited films. In addition, during crystallization, a number of defects in the structure of polycrystalline films are formed, which lead to an increase in leakage currents along grain boundaries [1]. This significantly limits the applicability of hafnium oxide in MOS structures. One of the methods for improving the properties and obtaining heat resistant amorphous HfO_2 films is based on doping hafnium oxide with impurities (e. g. Si, Al, Y, Gd, La, etc.) [8–12]. One of such materials is zirconium, since zirconium oxide ZrO_2 has great similarity to hafnium oxide in structural modification, chemical and physical properties and allows improving the heat resistance of HfO_2. Films of solid solutions of hafnium-zirconium oxide ($Hf_{1-x}Zr_xO_2$) have been widely studied and were initially considered exclusively as linear dielectrics [13]. Interest in them was due to the search for materials with high permittivity. In 2011, ferroelectric layers were obtained for the first time by doping HfO_2 with zirconium oxide ZrO_2, and it was found that the highest polarization was obtained for film composition $Hf_{0.5}Zr_{0.5}O_2$ [14]. This further increased interest in $Hf_{1-x}Zr_xO_2$ films.

One of the promising methods for forming multicomponent oxide films is the method of reactive magnetron sputtering of composite targets, i.e. targets consisting of a matrix of one metal with inserts of other metals [15]. This method allows obtaining multicomponent films with an arbitrary number and content of elements using one magnetron. However, to date, there are practically no publications that would analyze the features of the sputtering processes of composite targets during reactive sputtering. Thus, the aim of the work was to study the effect of zirconium doping on the dielectric properties of hafnium oxide films when using reactive magnetron sputtering of a composite target.

Experiment

The deposition of hafnium and hafnium-zirconium oxide films was performed by pulsed reactive magnetron sputtering of Hf and Hf-Zr targets in an Ar/O_2 working gas mixture. To deposit hafnium oxide films, a Hf target (99.9 % purity) ∅80 mm and 2 mm thick was used. To deposit hafnium-zirconium oxide films, a Hf-Zr composite target ∅80 mm and 2 mm thick was used, which consisted of two sectors of Hf (99.9 % purity) and Zr (99.9 % purity) in an area ratio of 10:12 (Fig. 1). The ratio of the target sector areas was selected based on the condition of depositing films with an atomic ratio of Zr and Hf in the film of 1:1, which corresponds to the region of formation of a solid solution $Hf_{1-x}Zr_xO_2$ with stable ferroelectric properties.



a *b*

Fig. 1. Scheme (*a*) and appearance (*b*) of Hf-Zr composite target

During the experiments, substrates made of highly doped n-type monocrystalline silicon Si(100) and polished optical quartz JGS-1 were mounted on a substrate holder at a distance of 120 mm from the magnetron target surface. The offset of the substrate holder axis relative to the magnetron axis was 100 mm. The substrate rotation rate was 24.5 rpm. The vacuum setup chamber was evacuated to a pressure of 10^{-3} Pa and the substrates were ion-cleaned. For this purpose, Ar was supplied to the ion source. The argon flow was 20 sccm. The cleaning time, voltage and discharge current were constant in all experiments and were t = 2 min, U_d = 90 V, I_d = 6.0 A, respectively. Then the films were deposited. For this purpose, a gas mixture (Ar/O_2) was supplied to the magnetron gas distribution system. The oxygen content in the chamber (G_{O2}) varied from 0 to 100 % with a total gas flow rate of 60 sccm. The pressure in the chamber was 0.08 Pa. The flow rate of the working gases was controlled using mass flow controller RRG-1. The magnetron was powered by a unipolar pulse current (pulse frequency F_p = 10 kHz, duty cycle D = 80 %). The magnetron discharge current was maintained constant in all experiments and was I_t = 1.5 A. The thickness of the deposited films was about 100 nm and was regulated by the deposition time. The thickness of the deposited films was determined using a POI-08 optical interferometric profilometer. The elemental composition of the deposited films was analyzed by energy-dispersive X-ray spectroscopy (EDX) using a Bruker QUANTAX 200 Energy Dispersive X-ray spectrometer attachment to a Hitachi S-4800 high-resolution field emission scanning electron microscope.

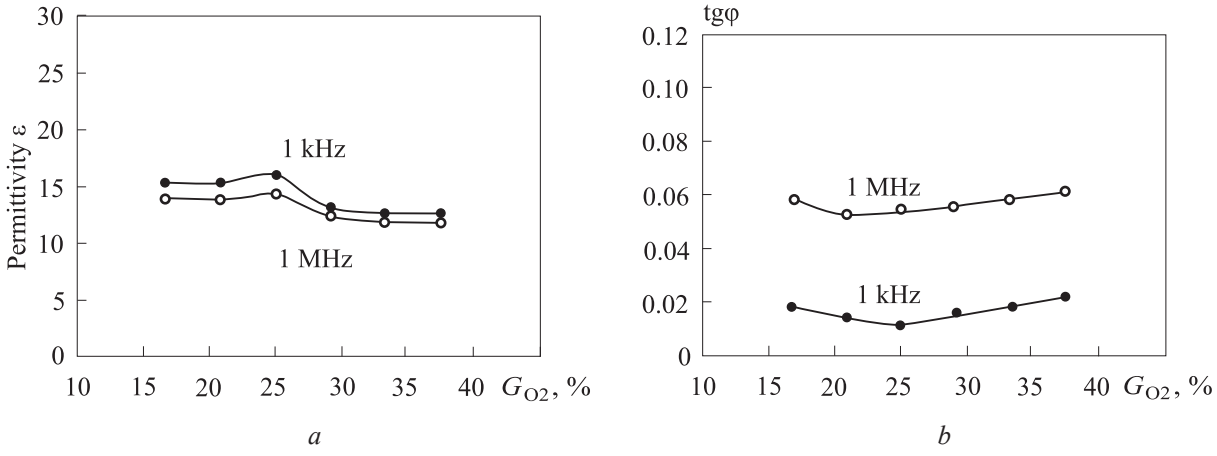
The dielectric characteristics of the deposited layers were measured on test MOS structures. For this purpose, the upper Ni electrode was deposited on the oxide film through a mask using the ion-beam sputtering method. The area of the upper capacitor electrode was 0.096 mm^2. The capacitance and dielectric loss tangent in the frequency range from 25 Hz to 1.0 MHz were obtained using an E7-20 immittance meter. The permittivity values were calculated based on the dielectric layer thickness and the capacitance of the capacitor structure. The frequency dispersion of the relative permittivity was calculated as the ratio of the ε values at a frequency of 25 Hz and 1.0 MHz

$$D_F = \frac{\varepsilon(25\ \text{Hz})}{\varepsilon(1.0\ \text{MHz})}.$$

To measure the current-voltage characteristics of the structures, a constant voltage was applied to the capacitor, changing from (–20) to 20 V, and the current flowing through the capacitor was recorded. The breakdown voltage of the MOS structures was recorded using a TR-4805 transistor characteristic tracer. During the measurements, a direct positive voltage was applied to the upper plate of the capacitor and increased from 0 to 50 V at a rate of 0.2 V/s. The breakdown voltage U_b was recorded by a sharp increase in the current through the capacitor with a current limit of 10 mA. During the research, all electrical measurements were made on 10 capacitors from the array and the obtained data were averaged. Optical transmission spectra in the range of 190–900 nm were obtained using a Proscan MS-121 spectrophotometer. The width of the band gap of the films was determined by the edge of their intrinsic optical absorption.

Results and discussion

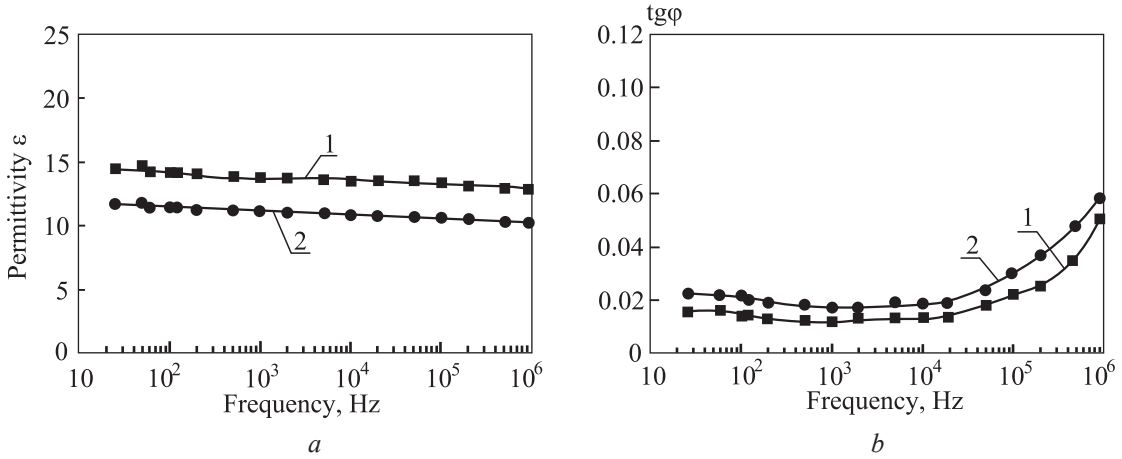
Fig. 2 shows the dependences of the permittivity and the dielectric loss tangent of the HfO_x films on the oxygen concentration in the Ar/O_2 gas mixture. The studies have shown that the formation of insulating films was observed at an oxygen concentration in the Ar/O_2 gas mixture of more than 12.5 %. At a lower oxygen concentration, the films were electrically conductive and had high values of the dielectric loss tangent (tgφ > 0.5).



a *b*

Fig. 2. Permittivity (*a*) and the dielectric loss tangent (*b*) of hafnium oxide films as a function of the oxygen concentration in the Ar/O_2 gas mixture

At G_{O2} more than 16.7 %, films with a permittivity from 12.5 to 16 were deposited (Fig. 2, *a*). In this case, tgφ was less than 0.02 at a frequency of 1 kHz and less than 0.06 at a frequency of 1 MHz (Fig. 2, *b*). It should be noted that when G_{O2} changed in the range from 16.7 to 37.5 %, the values of ε and tgφ were practically independent of the oxygen concentration. Analysis of the frequency dependences of the capacitance and dielectric loss tangent showed that the films deposited at an O_2 concentration of 16.7 to 37.5 % had low values of frequency dispersion (D_F = 1.15) and dielectric loss tangent over the entire frequency range (from 25 to 10^6 Hz) (Fig. 3). The leakage current density J_L of hafnium oxide films at zero bias was less than 10^{-5} A/m^2 (Fig. 4). In the films deposited at a low oxygen concentration (less than 12 % O_2), dielectric breakdown occurred at an electric field strength of $E = (3.0–5.0) \cdot 10^7$ V/m. For the films obtained at an oxygen concentration of 12.5–16.7 %, the leakage current smoothly increased with an increase in the bias voltage and reached a limit of 0.1 A/m^2 at $E = 5 \cdot 10^7$ V/m. At higher oxygen concentrations (over 20 %), the leakage currents decreased to $(1.0–3.0) \cdot 10^{-3}$ A/m^2 at $E = 5 \cdot 10^7$ V/m. The band gap of hafnium oxide films in the O_2 concentration range from 16.7 to 41.6 % remained practically the same and was 5.85–5.87 eV (Fig. 5, *a*). At lower oxygen contents, E_g decreased to 5.5 eV. The breakdown field strength for films deposited at an oxygen concentration of over 25 % was within the range of $(2.1–2.4) \cdot 10^8$ V/m (Fig. 5, *b*).



a *b*

Fig. 3. Frequency dependences of the permittivity (*a*) and the dielectric loss tangent (*b*) of HfO_x films deposited at different oxygen concentrations in an Ar/O_2 gas mixture: 1 – 20.8 %; 2 – 37.5 %

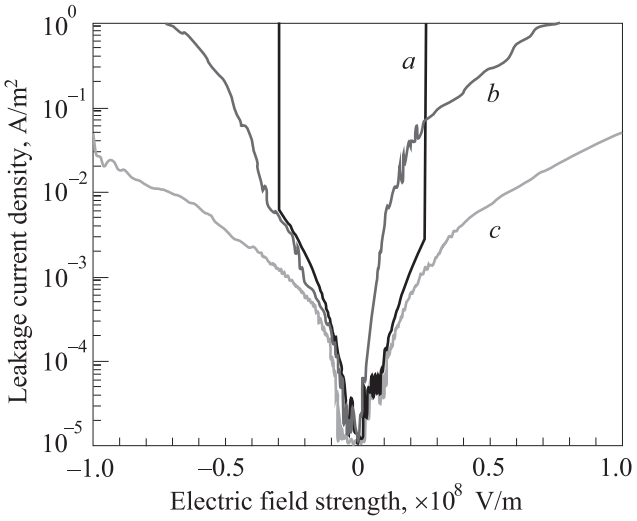


Fig. 4. Dependence of leakage current density on electric field strength for hafnium oxide films deposited at different oxygen concentrations in Ar/O_2 gas mixture: *a* – 9.0 %; *b* – 16.7 %; *c* – 26.0 %

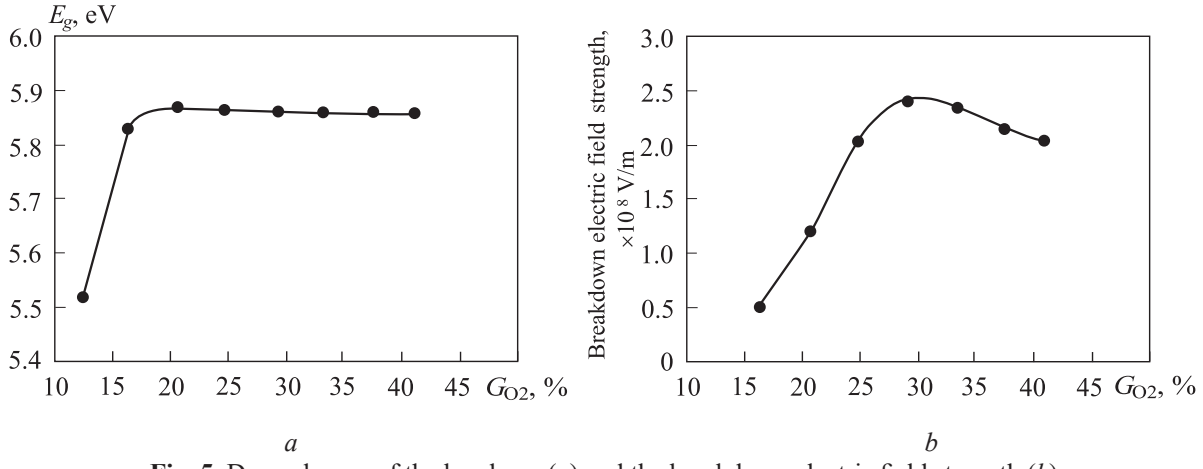


Fig. 5. Dependences of the band gap (*a*) and the breakdown electric field strength (*b*) of hafnium oxide films on the oxygen concentration in the Ar/O_2 gas mixture

The effect of zirconium doping on the dielectric characteristics of hafnium oxide films was also studied. To establish a correspondence between the sputtering process parameters, the elemental content in the films and their properties, the elemental composition of the films deposited by sputtering the Hf-Zr composite target was studied. The elemental content analysis by the EDX method showed that when sputtering the Hf-Zr composite target, the metal content in the films was C_{Zr} = 40.02 at.% and C_{Hf} = 59.98 at.%, respectively. Fig. 6 shows the frequency dependences of the permittivity and the dielectric loss tangent of the hafnium-zirconium oxide films deposited at different oxygen concentrations in the Ar/O_2 gas mixture. The insulating $Hf_{0.6}Zr_{0.4}O_y$ films were formed at G_{O2} greater than 12.5 %. With an increase in the oxygen concentration, a decrease in ε and dielectric losses was noted, especially at high frequencies. It should be noted that the films deposited at G_{O2} above 20 % were characterized by an extremely low frequency dispersion value (D_F less than 1.1).

Fig. 7 shows the dependences of the relative permittivity and the dielectric loss tangent of the $Hf_{0.6}Zr_{0.4}O_y$ films on the oxygen concentration in the Ar/O_2 gas mixture. When doped with zirconium, the permittivity of the hafnium oxide films remained practically the same. However, due to the decrease in frequency dispersion, a slight increase in ε was observed at high frequencies (Fig. 7, *a*). Dielectric losses at a frequency of 1 kHz were less than 0.01, and at a frequency of 1 MHz they decreased to 0.05–0.06. It should also be noted that ε and tgφ of the hafnium-zirconium oxide films remained practically the same when G_{O2} varied from 25 to 50 %.

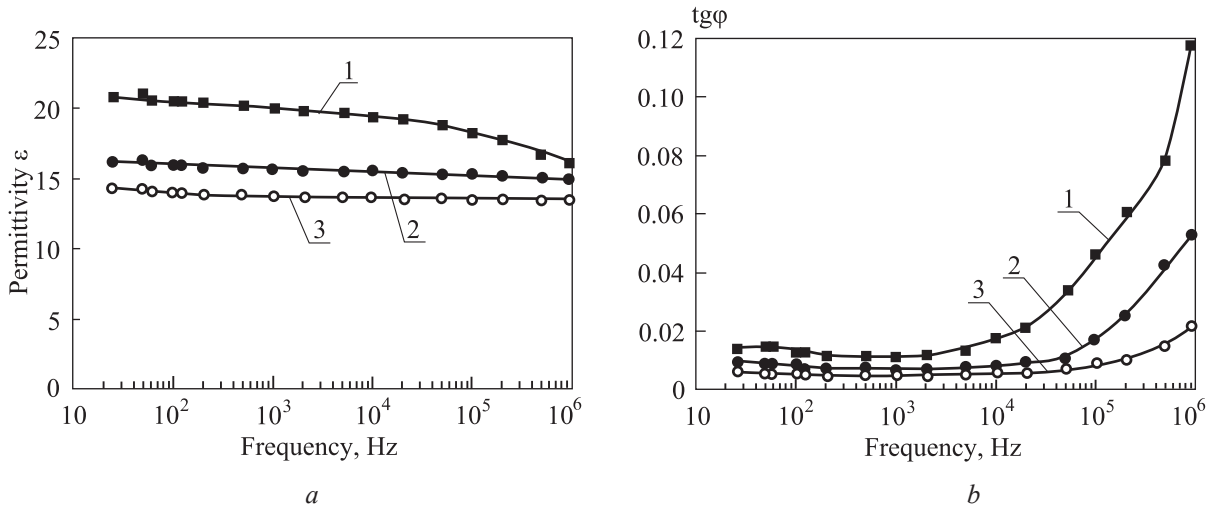


Fig. 6. Frequency dependences of the permittivity (*a*) and the dielectric loss tangent (*b*) of $Hf_{1-x}Zr_xO_2$ films deposited at different oxygen concentrations in the Ar/O_2 gas mixture: 1 – 12.5 %; 2 – 16.7 %; 3 – 29.2–41.7 %

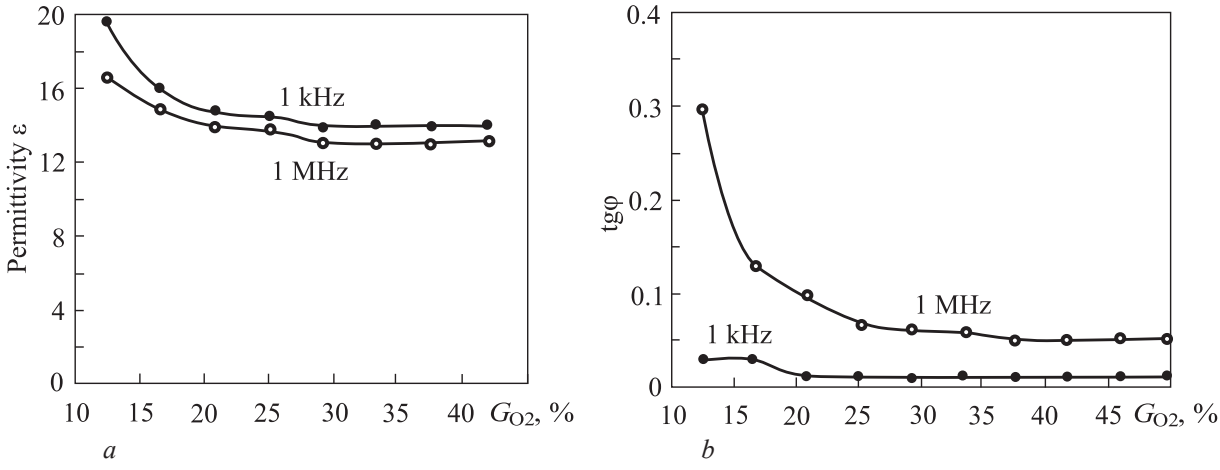


Fig. 7. Dependences of the permittivity (*a*) and the dielectric loss tangent (*b*) of hafnium-zirconium oxide films on the oxygen concentration in the Ar/O_2 gas mixture

Fig. 8 shows the dependences of the leakage current density on the electric field strength for hafnium-zirconium oxide films deposited at different oxygen concentrations in the Ar/O_2 gas mixture. The leakage current density of the $Hf_{0.6}Zr_{0.4}O_y$ films at zero bias for all samples deposited at G_{O2} above 12.5 % was less than 10^{-5} A/m^2. When a constant bias voltage was applied, the leakage current increased, and for the sample deposited at G_{O2} = 12.5 %, it was $2.0 \cdot 10^{-3}$ A/m^2 (Fig. 8, *a*). With an increase in the oxygen concentration in the Ar/O_2 gas mixture, the leakage current density at a constant bias voltage decreased, and at G_{O2} above 30 % (Fig. 8, *c*), films with $J_L = (3.0–5.0) \cdot 10^{-5}$ A/m^2 at $E = 5 \cdot 10^{-7}$ V/m were obtained.

Fig. 9 shows the dependences of the band gap and the breakdown field strength of $Hf_{0.6}Zr_{0.4}O_y$ films on the oxygen concentration in the Ar/O_2 gas mixture during film deposition. The band gap of the films (Fig. 9, *a*) at G_{O2} over 20 % was 5.87–5.89 eV and exceeded the E_g values of hafnium oxide. To compare, the band gap of hafnium oxides was 5.85–5.87 eV. The breakdown field strength of the films did not depend on the oxygen concentration and at G_{O2} over 16.7 % was $(2.5–3.0) \cdot 10^8$ V/m (Fig. 9, *b*).

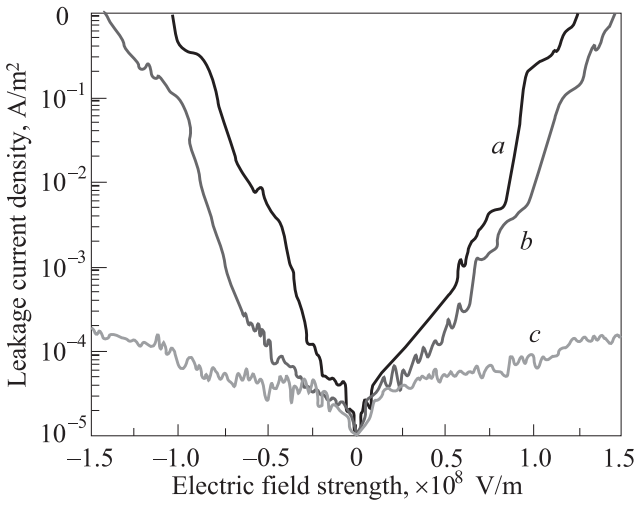


Fig. 8. Dependence of leakage current density on electric field strength for hafnium-zirconium oxide films deposited at different oxygen concentrations in Ar/O_2 gas mixture: *a* – 16.7 %; *b* – 25.0 %; *c* – 29.2–41.7 %

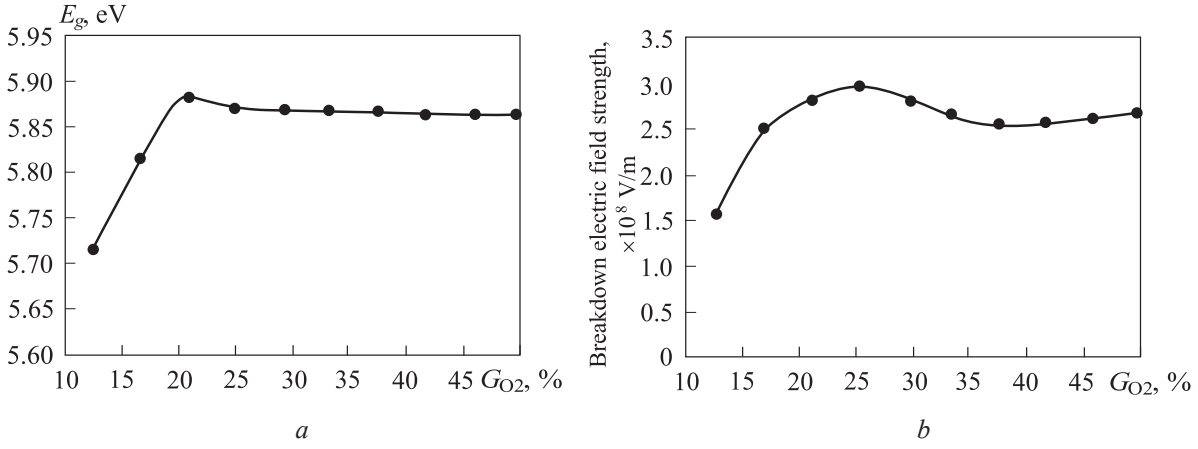


Fig. 9. Dependences of the band gap (*a*) and breakdown field strength (*b*) of hafnium-zirconium oxide films on the oxygen concentration in the Ar/O_2 gas mixture

Conclusion

1. The analysis of the obtained results shows that the method of pulsed reactive magnetron sputtering of a Hf target in an Ar/O_2 working gas mixture can be used to depositing hafnium oxide films with ε = 12.5–16.0 (F = 1 kHz), ε = 12–14 (F = 1 MHz), tgφ = 0.012–0.022 (F = 1 kHz) and tgφ = 0.05–0.06 (F = 1 MHz), E_g = 5.85–5.87 eV, $E_b = (2.1–2.4) \cdot 10^8$ V/m and relatively low leakage currents $J_L \approx (1.0–3.0) \cdot 10^{-3}$ A/m^2 at $E = 5 \cdot 10^7$ V/m. It should be noted that hafnium oxide films with good dielectric characteristics were formed in a relatively wide range of oxygen concentrations in the Ar/O_2 gas mixture (from 20.0 to 37.5 %) and the characteristics of the films were practically independent of G_{O2}. Doping of hafnium oxide with zirconium (40 at.%) allows to increase the permittivity from 12.5–16 to 14–16 at a frequency of 1 kHz and from 12–14 to 14–16 at a frequency of 1 MHz due to the reduction of frequency dispersion from D_F = 1.15 to values less than 1.1, to decrease the dielectric loss tangent to 0.008–0.012 at a frequency of 1 kHz and to 0.04–0.05 at a frequency of 1 MHz, to decrease the leakage current from $(1.0–3.0) \cdot 10^{-3}$ A/m^2 to $(3–5) \cdot 10^{-5}$ A/m^2 at $E = 5 \cdot 10^7$ V/m and to increase the breakdown voltage from $(2.1–2.4) \cdot 10^8$ V/m to $(2.5–3.0) \cdot 10^8$ V/m. At the same time, the band gap of the films remained practically the same and amounted to 5.87–5.89 eV. The obtained characteristics of the $Hf_{0.6}Zr_{0.4}O_y$ films are also observed in a relatively wide range of oxygen concentrations, which should ensure high reproducibility of the properties.

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