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Phonon-assisted electron tunneling between traps in silicon oxide films treated in hydrogen plasma

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A B S T R A C T

The charge transport in thin thermal silicon oxide films treated in electron cyclotron resonance hydrogen plasma at different exposure times was investigated. X-ray photoelectron studies show that such treatment leads to the oxygen deficiency of the films. It was established that the treatment of the films in plasma leads to an increase of their conductivity by a factor of about 10^2. The film charge transport properties were studied at different temperatures and analyzed within four theoretical dielectric conductivity models. It was found that the charge transport mechanism is described by Fowler-Nordheim model in the initial silicon oxide and by the model of phonon-assisted electron tunneling between neutral traps after the treatment in hydrogen plasma. The thermal trap ionization energy value (W_t = 1.6 eV) measured from transport experiments is in agreement with that obtained from *ab initio* calculations for the oxygen vacancy (Si-Si bond) in SiO_2.

1. Introduction

Oxygen-deficient silicon oxide thin films are favorable candidates for an active medium of resistive memory elements (memristors) with the operating characteristics based on the reversible switching of an oxide layer between high and low resistance states (Resistive Random Access Memory, RRAM) [1,2]. The switching of resistive states in such memristors is believed to be associated with the oxygen vacancies electro-diffusion which leads to the formation (or rupture) of a filament inside an oxide layer [3,4]. Nonstochiometric oxygen-deficient oxides including TaO_x, HfO_x, SiO_x and others [1,2,5] are considered to be the most attractive candidates applicable as an active medium in memristors. The competitive advantage of silicon oxide over other materials consists in its compatibility with the Si-based memory technology. One of the effective methods used for the formation of oxygen-deficient oxide thin films is the treatment of a stoichiometric oxide in electron cyclotron resonance (ECR) hydrogen plasma. This method was shown as well suitable for a partial reduction of HfO_2 [6] and V_2O_5 [7]. In particular, in Ref. [6], the treatment of HfO_2 in the hydrogen plasma was reported on as improving the switching characteristics of HfO_2-based memristors. This method has not been applied for SiO_2 yet.

The investigation of charge transport mechanism in dielectrics is one of the most sensitive methods to determine the microscopic parameters of traps responsible for conductivity including ionization energy and a capture cross-section, which, in turn, allows determining their nature. This method consists in the approximation of the experimental *j-F* characteristics by the ones simulated in the frames of different dielectric conductivity models and a comparison of fitting model parameters with theoretical or expected values. Such parameter as a trap ionization energy of a specified defect can be independently determined by the quantum-chemical modeling. The determination of charge transport mechanism and the nature of traps in dielectric films are of great importance on technological grounds as it is essential for the optimization of their synthesis conditions.

The aim of the present paper is to elucidate the possibility of generating high oxygen vacancies concentration in thermal SiO_2 films in a controllable manner by their treatment in ECR hydrogen plasma. We also aimed to investigate the possible charge transport mechanism of the treated silicon oxide films in the temperature range of 300-400 K. In addition, we aimed to reveal the nature of charge traps in these films, establishing the connection between the charge transport and high concentrations of oxygen vacancies, which is important for RRAM technology.

2. Experiment and data analysis

For the X-ray photoelectron spectroscopy (XPS) studies, a group of 3 samples with the p^{++}-Si/SiO_2 structure was prepared by the thermal oxidation of Si substrates with a further treatment in ECR hydrogen plasma during the varying times of 0, 6 and 14 min. The p^{++}-Si/SiO_2/

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Ni structures for measuring *j-F* characteristics were obtained in a similar manner with the additional step of Ni contacts deposition by the electron beam evaporation via a metallic mask with the windows sized 0.2×0.2 mm^2. These structures formed the second group of 4 samples (indicated as 1, 2, 3 and 4), the oxide layer of which had been treated in ECR hydrogen plasma during 0, 2, 6 and 14 min., respectively, before the top contact was deposited. For both groups of samples, the hydrogen pressure in a chamber, sample holder bias (-300 V) and the substrate temperature (538 K) were fixed during the plasma treatment. By means of a spectroscopic ellipsometer "Ellips-1771 SA" it was revealed that the increase of the treatment time leads to a monotonic decrease of the oxide film thicknesses with the values of 19.5, 18.7, 16.8 and 12.7 nm. The measurements of *j-F* characteristics at 300, 350 and 400 K were carried out with a Linkam LTS420E heated table and Keithley 6517a electrometer. To ensure the accuracy of the measured *j-F* curves at different temperatures, the samples were heated at the rate of 10 K per minute and, when they reached the desired temperature values, were kept at these values for no less than 10 minutes. At each temperature, the *j-F* curve measurements were repeated more than once and then the average *j-F* curves were obtained. The inaccuracy of the temperature measurements did not exceed ± 2 K. The DC current measurements error was less than ± 0.1 % at the highest current values and less than ± 1 % at the lowest ones.

The *j-F* characteristics were simulated in the frame of Fowler-Nordheim tunneling mechanism (for sample 1) and four possible temperature-dependent conductivity models, where the charge transport is limited by traps ionization (see Table 1). The Frenkel model [8,9] describes the thermal ionization of an isolated trap in an electric field reducing the trap Coulomb potential. According to the Hill-Adachi model [10,11], the barrier ionization energy is decreased due to the overlapping of neighboring Coulomb traps. In the case of Makram-Ebeid and Lannoo model [12], the charge transport is associated with the multi-phonon ionization of isolated charged traps. In the Nasyrov-Gritsenko model [13,14], the charge transfer occurs via the phonon-assisted tunneling between nearby neutral traps without their excitation in the conduction band.

The X-ray photoelectron spectra (XPS) were recorded on a VG ESCALAB HP electron spectrometer using the AlKα radiation ($h\nu$ = 1486.6 eV, 150 W). The [O]/[Si] atomic concentration ratios (parameter x) in the films were determined using the integral photoelectron peak intensities of silicon and oxygen after the subtraction of Shirley-type background and correction for the corresponding atomic sensitivity factors (ASF) [15]. The quantum-chemical modeling of the electronic structure of oxygen vacancies in SiO_2 (space group $P3_121$) was carried out in terms of the density functional theory using the hybrid exchange-correlation functional parameterization PBE0 [16] in the Quantum-ESPRESSO code. The cutoff energy of 75 Ry was used for plane waves, the core was described by norm-conserving pseudopotentials. Oxygen vacancies were simulated by removing oxygen atoms from a SiO_2 supercell of 72 atoms with a further structural relaxation. The thermal electron (hole) ionization energy E^{e}_{th} (hole E^{h}_{th}) of the defect was estimated by Eq. 1, which reflects the energy gain occurring when an electron (hole) is trapped onto a defect level [17,18].

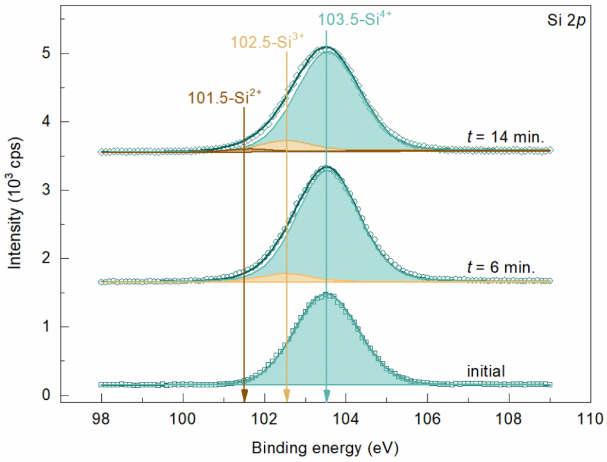

Fig. 1. Si 2*p* photoelectron spectra of silicon oxide films with different times of exposure to ECR hydrogen plasma (symbols). Solid lines represent the result of the experimental spectra decomposing into individual components. t is the duration of the film ECR hydrogen plasma treatment.

$$E^{e/h} = (E_p^{-1/+1} + E_d^0) - (E_p^0 + E_d^{-1/+1}) \quad (1)$$

Here E_p and E_d are ideal and defect supercell energies; the top index denotes the system charge.

3. Results and discussion

The analysis of the XPS spectra of Si 2*p* levels in silicon oxide films treated in ECR hydrogen plasma revealed that the peak width increases with the treatment time (Fig. 1). The full width at the half maxima of Si 2*p* peaks were 1.9, 2.0 and 2.05 for the samples treated in plasma for 0, 6 and 14 minutes, respectively. The recorded spectra were decomposed into individual components. The initial SiO_2 film (0 min. plasma treatment) spectrum was described by the peak at the binding energy of 103.5 eV, which corresponds to the Si^{4+} oxidation state. It was found that the plasma treatment leads to the appearance of additional peaks at the binding energies of 102.5 and 101.5 eV corresponding to Si^{3+} and

Table 1
Current density equations in different charge transport models in dielectrics. Here e and m_0 are electron charge and mass, m^* is the electron effective mass, Φ is the electron injection barrier height, $h = 2\pi\hbar$ is the Planck constant, k is the Boltzmann constant, ε_0 is vacuum permittivity, ε_∞ is the high-frequency relative permittivity, N is trap concentration, $s = N^{-1/3}$ is the average distance between traps, ν is the frequency factor, W_t and W_{opt} are thermal and optic trap ionization energies, W_{ph} is the phonon energy, F is the electric field.

Model	Current density equation
Fowler-Nordheim	$\frac{e^3 m_0}{8\pi\hbar m^* \Phi} F^2 \exp(-\frac{8\pi\sqrt{2em^*}}{3ehF}\Phi^{3/2})$
Frenkel	$eNs\nu \exp(-\frac{W-\beta_F\sqrt{F}}{kT})$, $\beta_F = \sqrt{\frac{e^3}{\pi\varepsilon_\infty\varepsilon_0}}$, $\nu = \frac{W}{h}$
Hill-Adachi	$2eNs\nu \exp(-\frac{W-\frac{e^2}{\pi\varepsilon_\infty\varepsilon_0 s}}{kT})\sinh(\frac{eFs}{2kT})$
Makram-Ebeid and Lannoo	$eNs\sum_{l=-\infty}^{+\infty}(\frac{lW_{ph}}{2kT} - \frac{W_{opt}-W_t}{W_{ph}}\coth(\frac{W_{ph}}{2kT}))I_n(\frac{W_{opt}-W_t}{W_{ph}\sinh(W_{ph}/2kT)})P_l(W_t + lW_{ph})$, $P_l = \frac{eF}{2\sqrt{2m^*W}}\exp(-\frac{4}{3}\frac{\sqrt{2m^*}}{\hbar eF}W^{3/2})$
Nasyrov-Gritsenko	$\frac{2eN\sqrt{\pi}\hbar W_t}{m^* s^2\sqrt{2kT(W_{opt}-W_t)}}\exp(-\frac{W_{opt}-W_t}{kT})\exp(-\frac{2s\sqrt{2m^*W_t}}{\hbar})\sinh(\frac{eFs}{2kT})$

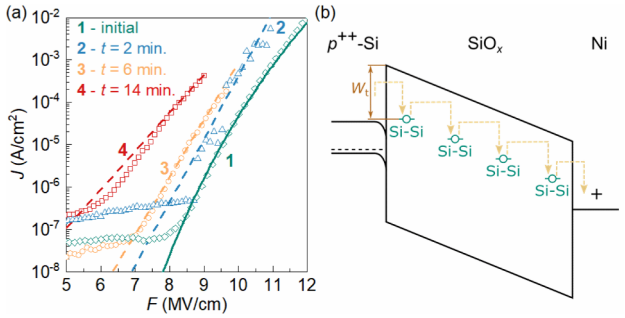

Fig. 2. (a) Experimental *j-F* curves of samples 1, 2, 3 and 4 (symbols). The solid line represents the *j-F* curve for sample 1 calculated in the Fowler-Nordheim model and dashed lines represent the *j-F* curves for samples 2-4 calculated in the phonon-assisted tunneling between traps model. *t* is the duration of the film ECR hydrogen plasma treatment. (b) Energy diagram illustrating the model of phonon-assisted tunneling between traps model in the investigated structure.

Si^{2+} oxidation states, respectively. This indicates the presence of oxygen vacancies in the treated films. The atomic ratio [O]/[Si], calculated from the XPS data, also indicates that the ECR plasma treatment of SiO_2 films leads to their oxygen depletion, as the *x* values were 2.0, 1.9 and 1.85 for the samples treated in plasma for 0, 6 and 14 min., respectively.

The experimental *j-F* characteristics of the initial stoichiometric SiO_2 film (sample 1) are well approximated by the theoretical ones calculated in the frames of the tunneling injection by Fowler-Nordheim mechanism with $m^* = 0.5\ m_0$ and $\Phi = 3.2$ eV (Fig. 2(a)). This result is predictable and in a good agreement with the known data [19]. The *j-F* characteristics of samples 2, 3 and 4 shown in the same figure depict that the silicon oxide film conductivities increase after the treatment of silicon oxide in hydrogen plasma, and the conductivity values increase stronger at longer treatment times. Sample 4 demonstrates that the conductivity is increased by to 2 orders of magnitude in comparison with that of the sample with the initial film. This can be explained by the insertion of structural defects of high concentration into a SiO_2 film.

For revealing the nature of these defects, the conductivity mechanism of the samples treated in plasma was investigated. For this purpose, the experimental *j-F* characteristics at different temperatures for one of the samples (sample 3) were analyzed using the above-mentioned charge transport models. As can be seen in Fig. 3, the experimental *j-F* characteristics can be formally described by each model. However, the Frenkel model describes the experimental *j-F* characteristics at the acceptable values of $W = 1.4$ eV, $\nu = W/h = 3.4\times10^{14}\ s^{-1}$ and $\varepsilon_\infty = 2.5$, while the trap concentration of $N_t = 1.0\times10^{1}\ cm^{-3}$ is anomaly small (the typical value of traps in dielectrics is in the range of $10^{18}-10^{22}\ cm^{-3}$) [14] (Fig. 3(a)). The Hill-Adachi model describes the experimental data with the reasonable values of $W = 2.13$ eV, $N_t = 1.5\times10^{20}\ cm^{-3}$ and $\varepsilon_\infty = 2.3$; meanwhile, the frequency factor ν takes the unphysically small value of $6.0\times10^{1}\ s^{-1}$ (Fig. 3(b)). Using the Makram-Ebeid and Lannoo model the best match of calculated and experimental data occurs at $W_t = 2.0$ eV, $W_{opt} = 4.0$ eV, $W_{ph} = 70$ meV and $m^* = 0.15\ m_0$, while the trap concentration $N_t = 2.0\times10^{13}\ cm^{-3}$ is rather low and, moreover, the model does not describe the *j-F* curve measured at 300 K (Fig. 3(c)).Therefore, it can be concluded that Frenkel, Hill-Adachi, and Makram-Ebeid and Lannoo models failed to describe the charge transport mechanism in silicon oxide films treated in electron cyclotron hydrogen plasma.

Only the Nasyrov-Gritsenko model consistently represents the experimental *j-F* characteristics of SiO_x, which means that the values of all model parameters $W_t = 1.6$ eV, $W_{opt} = 3.2$ eV, $m^* = 1.4\ m_0$ and $N_t = 1.6\times10^{20}\ cm^{-3}$ ($s = 1.84$ nm) are physically adequate (see Fig. 3(d)). The increased effective mass value, in comparison with the value for thermal SiO_2 ($m^* = 0.5\ m_0$), can be explained by the presence of the space charge of high traps concentration in the oxide film. A similar feature was observed when describing the current-voltage characteristics by Nasyrov-Gritsenko model for HfO_2 films and the organosilicate low-κ dielectric [20,21]. Taking into account the space charge using Poisson equation is quite a challenging task for numerical calculations and is beyond the scope of the present research.

The Nasyrov-Gritsenko model also well describes the *j-F* characteristics for the samples with longer (sample 2) and shorter (sample 4) treatment times in plasma using the same trap parameters values $W_t = 1.6$ eV, $W_{opt} = 3.2$ eV (see Fig. 2(a)). It was found that the increase of treatment time in plasma leads to the monotonic increase of traps concentration: for samples 2, 3 and 4 - $N_t = 18\times10^{19}$, 24×10^{19} and $80\times10^{19}\ cm^{-3}$, respectively. The values of m^* also differ (1.95 m_0, 1.88 m_0 and 1.75 m_0, respectively), which can be explained by the difference of the space charge influence depending on N_t and dielectric thickness. The *j-F* characteristics of the sample 3 presented in Fig. 2 and 3 were measured on different contacts. One can notice (Fig. 2 and 3) that the experimental and simulated $j-F$ curves diverge at small electric fields. This discrepancy is caused by the fact that at such electric field values the current is limited by the charges contact injection from electrodes into traps in the dielectric and, therefore, it is no longer limited by the Nasyrov-Gritsenko mechanism. The values of W_t and W_{opt} well coincide with the same values of 1.7 ± 0.2 eV and 3.0 ± 0.5 eV obtained earlier in the experiments based on the thermo- and photodepolarization of defects [22]. It should be mentioned that, in Ref. [22], the oxygen vacancies were assumed to be electron traps in SiO_2.

To confirm the hypothesis that it is oxygen vacancies (Si-Si bounds) that act as traps in the silicon oxide depleted with oxygen after the

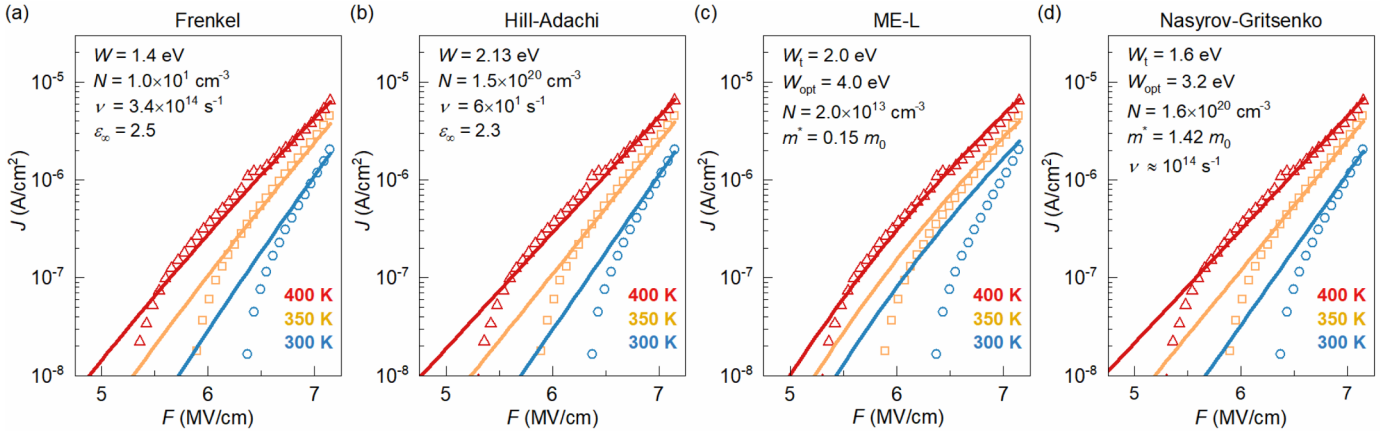

Fig. 3. Experimental (symbols) and simulated (lines) *j-F* curves of sample 3 at different temperatures. The simulated curves were calculated in Frenkel (a), Hill-Adachi (b), Makram-Ebeid and Lannoo (c) and Nasyrov-Gritsenko (d) models. The parameter values used for each model are indicated above the *j-F* curves.

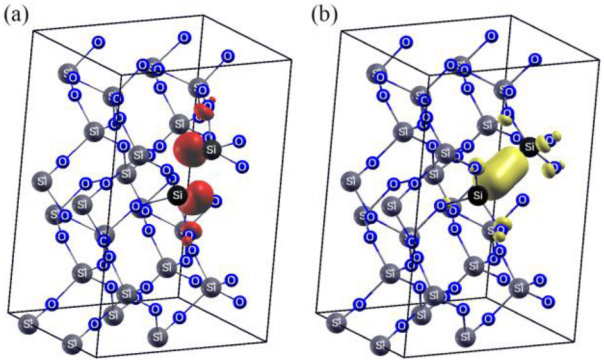

Fig. 4. Spacial localizaton of an electron (a) and a hole (b) added to the 72-atom α-SiO_2 supercell with oxygen vacancy.

hydrogen plasma treatment, *ab initio* calculations of the oxygen vacancies electronic structure in SiO_2 were carried out. It was shown that the localization of both electron and hole on a Si-Si bond is energetically favorable. The charge density of electron and hole added to a defective supercell is distributed on and between the atoms of the Si-Si bond (see Fig. 4), respectively. It is generally known that a hole localization on a Si-Si bond undergoes with the formation of the E'-center [23,24]. Only indirect experimental data [22,25] prove the electron localization.

The trap thermal ionization energy values of the oxygen vacancy calculated by Eq. 1 for electrons and holes are equal to 1.2 eV and 1.6 eV, respectively. It should be noted that the current approach regularly underestimates the electron trap energy due to the necessity of using the compensation background charge in calculations of periodic charged systems. As a rule, the error of the method is of the order of 0.1 eV [26]. Thus, the *ab initio* calculated thermal energy value for the oxygen vacancy is in agreement with that of the trap thermal energy of 1.6 eV for silicon oxide films treated in hydrogen plasma. Therefore, the charge transport in SiO_2 films treated in ECR hydrogen plasma is governed by the mechanism of phonon-assisted electron tunneling between neighboring traps, the role of which is fulfilled by oxygen vacancies (Si-Si bonds). A schematic illustration of this mechanism is presented in Fig. 2(b).

Amorphous SiO_2 is composed of $\equiv Si-O-Si\equiv$ structural units. The formation of oxygen vacancies ($\equiv Si-Si\equiv$), when treating SiO_2 in hydrogen plasma, can be described by the following scheme:

$$\equiv Si{-}O{-}Si \equiv + H^+ \rightarrow \equiv Si - Si \equiv + OH^+ \quad (2)$$

According to this scheme, an electron from oxygen migrates to the proton reducing the energy of the Si–O bond, which results in the formation of an oxygen vacancy and an OH^+ group. The similar mechanism for SiO_2 was earlier described in Ref. [27].

4. Conclusions

Thus, it was established that the treatment of thermal SiO_2 films in ECR hydrogen plasma results in the generation of high structural defects concentration (from 10^{19} to 10^{21} cm^{-3}). According to the photoelectron spectroscopy, such treatment leads to the oxygen deficiency of the films, as indicated by the presence of a mixture of multiple Si oxidation states and the decrease of the atomic ratio [O]/[Si] from 2.0 to 1.85. After the plasma treatment, the charge transport mechanism changes from the contact-limited Fowler-Nordheim tunneling injection to the bulk-limited trap-limited transport. It was shown that the charge transport in oxygen-deficient silicon oxide films can be consistently described by the Nasyrov–Gritsenko model of phonon-assisted tunneling between traps. The values of thermal and optical traps ionization energies were determined as W_t = 1.6 eV and W_{opt} = 3.2 eV, respectively. Based on the quantum-chemical modeling it was shown that oxygen vacancies (Si-Si bonds) represent the traps participating in the charge transport. It proves that the SiO_2 treatment in hydrogen plasma leads to the generation of oxygen vacancies in it. The proposed method of obtaining oxygen-deficient films can be optimized to provide further developments and the optimization of SiO_x-based RRAM elements.

CRediT authorship contribution statement

V.A. Voronkovskii: Investigation, Formal analysis, Visualization, Writing - original draft. **T.V. Perevalov:** Formal analysis, Visualization, Writing - review & editing. **R.M.H. Iskhakzay:** Methodology, Investigation. **V.Sh. Aliev:** Methodology, Supervision. **V.A. Gritsenko:** Conceptualization, Supervision, Validation. **I.P. Prosvirin:** Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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