

9

Research Article

Effect of low-energy electron irradiation on voltagecapacity curves of $Al/SiO_2/Si$ structure

Yuriy O. Kulanchikov¹, Pavel S. Vergeles¹, Eugene B. Yakimov¹

1 Institute of Microelectronics Technology and High-Purity Materials of the Russian Academy of Sciences, 6, Academician Ossipyan Str., Chernogolovka, 142432, Russia

Corresponding author: Pavel S. Vergeles (vergelesp@gmail.com)

Received 29 October 2019 • Accepted 18 December 2019 • Published 31 December 2019

Citation: Kulanchikov YuO, Vergeles PS, Yakimov EB (2019) Effect of low-energy electron irradiation on voltage-capacity curves of Al/SiO₂/Si structure. Modern Electronic Materials 5(4): 175–179. https://doi.org/10.3897/j.moem.5.4.52311

Abstract

Charging of dielectric targets by electron irradiation is a well-known phenomenon which should be taken into account in characterization of dielectric materials and coatings with electron microscopy, in electron beam lithography, in development of dielectric coatings for spacecrafts and other fields of science and engineering. Charging kinetics is strongly affected by spatial distribution of electrons and holes formed by irradiation. At the experimental electron beam energy electron penetration depth is smaller than dielectric thickness and this allows identifying the contribution of excess carrier transport to trap formation at the SiO₂/Si interface. Low-energy electron beams have been shown to substantially affect C-V curve slope, i.e., to form traps at the interface. We have studied the effect of bias applied to the test structure before and after electron beam irradiation. The experiment has shown that bias of either polarity applied to the test MOS structure before low-energy electron beam irradiation practically does not affect the C-V curves of the test structure. Positive bias applied to the metallization layer during low-energy electron beam irradiation has a strong effect on the C-V curve pattern while negative bias affects the C-V curves of the test structure restore slowly even at room temperature. Application of negative bias decelerated charge relaxation.

Keywords

silicon oxide, low-energy electron beam, MOS structure, C-V curves

1. Introduction

Charging of dielectric targets by electron irradiation is a well-known phenomenon which should be taken into account in studies of dielectric materials and coatings with electron microscopy, in electron beam lithography, in development of dielectric coatings for spacecrafts and in other fields of science and engineering. Most of the works reported so far dealt with measuring the potential of the charged surface which is mainly determined by the balance between the charge of the primary electrons penetrating into the material and the charge of the emitted secondary electrons [1–9]. It was shown however [10] that charging kinetics is strongly affected by the spatial distribution of the electrons and holes formed by irradiation. This distribution determines the effect of electron beam irradiation on the parameters of the metal–oxide–semiconductor (**MOS**) structures because the charge at the surface is compensated by the charge of the metallic contact.

The aim of this work is to study the effect of electron beam irradiation on the parameters of Al/SiO₂/Si structures. The study included measurements of voltage-ca-

© 2019 National University of Science and Technology MISiS. This is an open access article distributed under the terms of the Creative Commons Attribution License (CC-BY 4.0), which permits unrestricted use, distribution, and reproduction in any medium, provided the original author and source are credited. pacity curves (*C*–*V*) that are sensitive to the charge at the SiO_2/Si interface and in the bulk of the SiO_2 film near the SiO_3/Si interface.

2. Experimental

The effect of electron beam irradiation on the parameters of the test MOS structure was studied on boron doped Si substrates of *p*-type conductivity with an impurity concentration of 3×10^{14} cm⁻³ and a SiO₂ dielectric layer thickness of 200 nm. The oxide layer was produced by thermal oxidation of silicon. The diameter of the metallic pads was 1.6 mm, however the actual contact areas could decrease as a result of multiple measurements with a spring probe.

The capacitance-voltage curves were measured with a PAR Model 410 *C*–*V* plotter at 1 MHz. The irradiating electron beam parameters were as follows: an acceleration voltage of 2.5 kV, a beam current of within 1 nA and irradiation doses of 10, 20, 25 and 30 μ Cl/cm². The specimens were irradiated in a Jeol-840A electron microscope in TV mode through deposited Al metallization. The specimens were earthed in all the experiments and therefore the charge accumulated in SiO₂ was compensated by the charge at the metallic contact. Under these irradiation conditions the incident electrons did not reach the SiO₂/Si interface because the electron penetration depth into the oxide layer was within 80 nm.

We also studied the effect of bias applied during electron beam irradiation on the charge accumulation. The irradiation parameters were the same as in the previous experiments but positive or negative bias was applied to the metallization for moving the semiconductor surface at the SiO_2/Si interface either to accumulation or to strong inversion, respectively. The bias was produced by a B5-49 DC source.

3. Results and discussion

Figure 1 shows typical C-V curves before and after electron beam irradiation. Comparison with a calculated ideal curve showed that the C-V curve of the non-irradiated structure is already shifted towards negative voltages but the curve slope is close to that of the ideal one. This shift of the C-V curve can be accounted for by the combined effect of two components. One component is the difference in the work functions of the metal and the dielectric which is about -1 V. However since the shift of the initial C-V curve of the test structure is far greater than -1 V the main contribution to the C-V curve shift must come from the other component, i.e., the built-in positive charge in the bulk of the SiO₂ dielectric layer. Low-energy electron irradiation changes the slope of C-V curve but does not cause any substantial shift towards negative voltages. The change of the C-V curve slope is usually accounted for by the formation of traps at the Si/SiO₂ interface [11, 12]. However at the electron beam parameters used in this

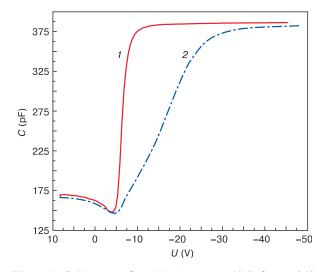


Figure 1. *C*–*V* curves of test MOS structure (*1*) before and (*2*) after irradiation. Acceleration voltage is of 2.5 keV, beam current of 1 nA and irradiation dose of 15 μ Cl/cm².

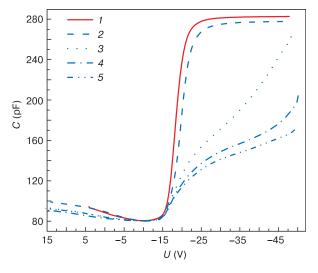


Figure 2. *C*–*V* curve variation for different irradiation doses: (*1*) before irradiation and (2–5) irradiation with doses of 10, 20, 25 and 30 μ Cl.

work, primary electrons do not reach the Si/SiO_2 interface and therefore trap formation at the Si/SiO_2 interface can only be accounted for by charge transport in the oxide layer bulk and/or carrier exchange with the silicon substrate.

Then we studied the effect of irradiation dose on the C-V curves of the test structure. The results are shown in Fig. 2. The lower maximum capacitance of the structure in this experiment is accounted for by the smaller metallic contact area. It can be seen that an increase in the irradiation dose reduces the slope of the C-V curve while the voltage shift in the inversion region practically does not depend on irradiation. Bulk charge in dielectric films is usually determined from voltage shift for flat zones in comparison with the calculated ideal curve [13] and a change in the curve slope indicates the formation of surface states. Then however a negligible shift of the C-V

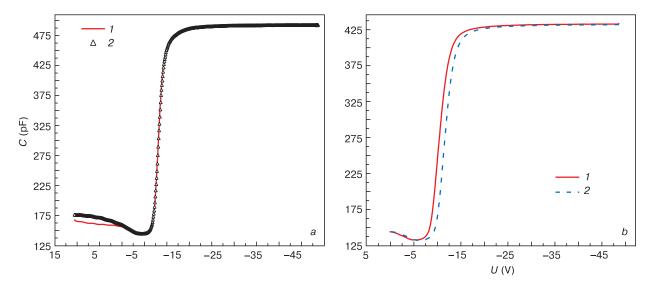


Figure 3. Variation of C-V curves of test MOS structure before electron beam irradiation for application of different bias to metallization (a) +40 V (inversion), (b) -40 V (accumulation), (1) before biasing and (2) after biasing for 15 min.

curve in the inversion region suggests that the effect of volume charge exactly compensates the effect of surface states. It is more plausible to assume that low-energy electron beam irradiation only slightly changes the bulk charge in the SiO₂ film whereas the density of states at the Si/SiO₂ interface increases with an increase in the irradiation dose.

To verify the assumption of carrier exchange between the oxide and the silicon substrate we studied the effect of bias applied to the metal contact on charge accumulation during irradiation and subsequent charge relaxation. We first verified the effect of bias applied to the initial MOS structure at room temperature. Figure 3 shows results of this experiment for bias voltages of -40 V and +40 V. It can be seen from Fig. 3a that positive bias does not affect the built-in charge in the dielectric. Negative bias slightly shifts the curve towards negative voltages without changing the curve slope (Fig. 3b). This shift indicates an increase in the positive charge in the bulk of the oxide layer and/or its shift toward the interface. Since negative bias application impedes the shift of the positive charge toward the interface the most probable cause of the C-Vcurve shift is hole injection from silicon to SiO₂.

Study of the effect of bias applied to metallization on charge accumulation during electron beam irradiation revealed the following regularities. The effect of irradiation is smaller if a negative bias is applied to the metal contact (the electric field in the dielectric attracts holes to the metal contact pad) than for a positive bias (the electric field in the dielectric repels holes to the Si/SiO₂ interface). Figure 4 shows C-V curves of the same metal contact to which bias was applied during irradiation (the irradiation dose was 20 μ Cl/cm²). It can be seen from Fig. 4 that application of a positive bias of 10 V to the metallization not only changes the C-V curve slope but also shifts the curve towards negative voltages. One can therefore conclude that charge is accumulated not only at the Si/SiO₂ interface but also in the dielectric bulk.

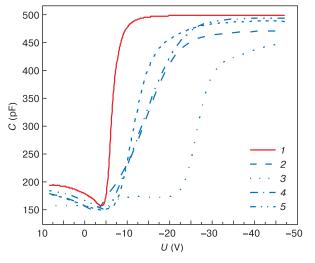


Figure 4. Variation of C-V curves of test MOS structure (1) before irradiation and (2–5) after irradiation with different bias: (2) +10 V, (3) 0, (4) –10 V and (5) –20 V. Irradiation dose: 20 µCl/cm².

As noted above the depth of electron/hole pair generation for the electron beam energy used in this experiment is within one half of the SiO, film thickness. Moreover it was shown earlier [14, 15] that the hole free path in SiO is a few tens of nanometers. It should be also noted that the built-in electric field in the unirradiated specimen generated by the positive charge in the oxide layer suppresses hole transport toward the Si/SiO, interface and stimulates electron drift toward the interface. The contribution of the charges near the metal contact to this field is screened and hence the main contribution to the field comes from the positive charge at the Si/SiO, interface. Our experimental results show that irradiation of the structure at a zero bias increases the positive charge in the oxide layer. The electric field inside the dielectric should impede nonequilibrium hole transport toward the interface and stimulate

electron transport toward the interface. Thus our experimental results suggest that the formation of surface states at the Si/SiO₂ interface due to low-energy electron irradiation of the structure is stimulated by nonequilibrium electrons reaching the interface. This however does not agree with the common opinion [11, 12] that nonequilibrium electrons leave rapidly the specimen or recombine inside it and the formation of surface states and bulk charge involve non-recombined nonequilibrium holes. Noteworthy, our study of the effect of electric field application during irradiation shows that nonequilibrium holes can also stimulate the formation of surface states (Fig. 4) although in this case the process can be stimulated by electron injection from silicon which is known to cause MOS structure device degradation [16–19].

It was also of interest to study the stability of the irradiation induced charge. The study showed that C-V curve recovery occurs even at room temperature, though very slowly. Charge relaxation was found to obey a logarithmic law

$$\Delta V = \Delta V_0 - A \ln(t/t_0),$$

where ΔV_0 is the bias applied after electron beam irradiation measured at 0.8–0.9 of the capacity in the enriched region, t is the annealing time, A is the coefficient equal to 0.1–1 V and t_0 is the normalization constant equal to approx. 150 s. According to earlier works [20, 21] the logarithmic dependence can be accounted for by electron tunneling from the silicon substrate to the SiO₂ layer and compensation of the positive charge accumulated as a result of electron beam irradiation. To confirm this assumption we studied the effect of bias applied to the metallic

References

- Jbara O., Belhaj M., Odof S., K. Msellak, Rau E.I., Andrianov M.V. Surface potential measurements of electron-irradiated insulators using backscattered and secondary electron spectra from an electrostatic toroidal spectrometer adapted for scanning electron microscope applications. *Rev. Sci. Instrum.*, 2001; 72(3): 1788–1795. https://doi. org/10.1063/1.1344596
- Cazaux J. Scenario for time evolution of insulator charging under various focused electron irradiations. J. Appl. Phys., 2004; 95(2): 731–742. https://doi.org/10.1063/1.1632015
- Di Santo G., Coluzza C., Flammini R., Zanoni R., Decker F. Spatial, energy, and time-dependent study of surface charging using spectroscopy and microscopy techniques. J. Appl. Phys., 2007: 102(11): 114505. https://doi.org/10.1063/1.2817915
- Jbara O., Fakhfakh S., Belhaj M., Rondot S., Hadjadj A., Patat J.M. Charging effects of PET under electron beam irradiation in a SEM. J. Phys. D: Appl. Phys., 2008; 41(21): 245504. https://doi. org/10.1088/0022-3727/41/24/245504
- Cornet N., Goeuriot D., Guerret-Piécourt C., Juvé D., Tréheux D., Touzin M., Fitting H.-J. Electron beam charging of insulators with surface layer and leakage currents. *J. Appl. Phys.*, 2008; 103(6): 064110. https://doi.org/10.1063/1.2890427

electrode on the relaxation of the charge accumulated after irradiation. The study showed that the accumulated charge relaxation at room temperature was faster for positive bias than for negative bias. This can be accounted for by the fact that in inversion mode (positive bias) more electrons can tunnel to the SiO_2 layer from the silicon substrate whereas in accumulation (negative bias) electron injection into the oxide layer is largely suppressed.

4. Conclusion

The effect of low-energy electron beam irradiation on the C-V curves of a Al/SiO₂/Si MOS structure was studied for a ground metallic contact and for bias application during irradiation. The results suggest that at the electron energy used in the experiment for irradiation, the formation of surface states at the Si/SiO₂ interface can be stimulated not only by nonequilibrium holes as was commonly believed earlier but also by nonequilibrium electrons. The thermal stability of the bulk charge and surface states induced by irradiation was studied. The study showed that annealing of the irradiated MOS structure at 210 °C leads to an almost complete recovery of the initial state of the MOS structure.

Acknowledgments

The work of P. S. Vergeles and Yu. O. Kulanchikov was supported by RFBR grant No. 18-32-00323.

- Fitting H.-J., Meyza X., Guerret-Piécourt C., Dutriez C., Touzin M., Goeuriot D., Tréheux D. Selfconsistent electrical charging in insulators. *J. Europ. Ceramic Soc.*, 2005; 25(12): 2799–2803. https://doi. org/10.1016/j.jeurceramsoc.2005.03.143
- Belhaj M., Jbara O., Filippov M.N., Rau E.I., Andrianov M.V. Analysis of two methods of measurements of surface potental of insulators in SEM: electron spectroscopy and X-ray spectroscopy methods. *Appl. Surf. Sci.*, 2001; 177(1–2): 58–65. https://doi.org/10.1016/ S0169-4332(01)00209-4
- Rau E.I., Tatarintsev A.A., Kupreenko S.Y., Zaitsev S.V., Podbutsky N.G. Comparative analysis of methods for surface potential measurement of dielectrics charging under electron beam irradiation in scanning electron microscope. *Poverkhnost'. Rentgenovskie*, *sinkhronnye i neitronnye issledovaniya*, 2017; (10): 69–76. (In Russ.). https://doi.org/10.7868/S0207352817100110
- Rau E.I., Tatarintsev A.A., Zykova E.Y., Ivanenko I.P., Kupreenko S.Y., Minnebaev K.F., Khaidarov A.A. Electron-beam charging of dielectrics preirradiated with moderate-energy ions and electrons. *Phys. Solid State*, 2017; 59(8): 1526–1535. https://doi.org/10.1134/S1063783417080212
- Rau E.I., Evstafyeva E.N., Andrianov M.V. Mechanisms of charging dielectrics when they are irradiated with medium-energy electron

beams. Fizika tverdogo tela, 2008; 50(4): 599–607. https://doi. org/10.1134/S1063783408040057 (In Russ.)

- Oldham T.R., McLean F.B. Total ionizing dose effects in MOS oxides and devices. *IEEE Trans. Nucl. Sci.*, 2003; 50(3): 483–499. https://doi.org/10.1109/TNS.2003.812927
- Schwank J.R., Shaneyfelt M.R., Fleetwood D.M., Felix J.A., Dodd P.E., Paillet P., Ferlet-Cavrois V. Radiation effects in MOS oxides. *IEEE Trans. Nucl. Sci.*, 2008; 55(4): 1833–1853. https://doi. org/10.1109/TNS.2008.2001040
- Schroder D.K. Semiconductor materials and device characterization. Hoboken (New Jersey): John Wiley & Sons, Inc., 2006, 781 p.
- Borisov S.S., Vergeles P.S., Yakimov E.B. Investigations of electron beam induced conductivity in silicon oxide thin films. *Poverkhnost'. Rentgenovskie, sinkhronnye i neitronnye issledovaniya*, 2010; (9): 62–66. (In Russ.)
- Glavatskikh I.A., Kortov V.S., Fitting H.-J. Self-consistent electrical charging of insulating layers and metal-insulator-semiconductor structures. J. Appl. Phys., 2001; 89(1): 440–448. https://doi. org/10.1063/1.1330242
- Groeseneken G., Bellens R., Van den Bosch G., Maes H.E. Hot-carrier degradation in submicrometre MOSFETs: from uniform injection

towards the real operating conditions. *Semicond. Sci. Technol.*, 1995; 10(9): 1208–1220. https://doi.org/10.1088/0268-1242/10/9/002

- Acovic A., La Rosa G., Sun Y.-C. A review of hot-carrier degradation mechanisms in MOSFETs. *Microelectr. Reliab.*, 1996; 36(7–8): 845–869. https://doi.org/10.1016/0026-2714(96)00022-4
- Vuillaume D., Bravaix A., Goguenheim D. Hot-carrier injections in SiO₂. *Microel. Reliab.*, 1998; 38(1): 7–22. https://doi.org/10.1016/ S0026-2714(97)00179-0
- Cho M., Roussel P., Kaczer B., Degraeve R., Franco J., Aoulaiche M., Chiarella T., Kauerauf T., Horiguchi N., Groeseneken G. Channel hot carrier degradation mechanism in long/short channel n-Fin-FETs. *IEEE Trans. Electron Dev.*, 2013; 60(12): 4002–4007. https:// doi.org/10.1109/TED.2013.2285245
- Lelis A.J., Oldham T.R., Boesch H.E., McLean F.B. The nature of the trapped hole annealing process. *IEEE Trans. Nucl. Sci.*, 1989; 36(6): 1808–1815. https://doi.org/10.1109/23.45373
- Schmidt M., Köster (Jr) H. Hole trap analysis in SiO₂/Si structures by electron tunneling. *Phys. Stat. Sol. (b)*, 1992; 174: 53–66. https:// doi.org/10.1002/pssb.2221740106