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USE OF NANO-DIMENSIONAL HYDROPHOBIC COATINGS FOR OBTAINING ELECTRETS BASED ON SILICON DIOXIDE

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The article considers the physical-technological foundations of formation of the silicon dioxide (SiO_2) based electret for use in devices of MEMS technology. Studies have shown that the best electret properties are in SiO_2 obtained in «wet» oxygen medium as compared to samples obtained by other oxidation methods. This is probably due to the large number of Si-OH groups on the surface of the oxide in the «wet» SiO_2, which increases the effectiveness of the hydrophobic coatings during the modification of the SiO_2 surface. It has been found that other methods of obtaining oxide, for example, electrochemical or plasmachemical, do not make it possible to obtain SiO_2 with good electret properties. The decrease of the charge injected into an electret can occur due to the presence of volume or surface conductivity, as well as the screening of this charge by opposite charges from the medium, leading to significant decrease of electret surface potential at high ambient humidity. To increase the stability of the electret effect, it is necessary to perform water-repellency treatment of SiO_2 surface by applying thin (nanosized) water-repellent coatings. Experimental results on the stability of the electret surface potential are presented for usage of various water repellents. The most promising water repellents are high-temperature photoresist FPT-1-40 and polyimide nanolayer compositions – Langmuir-Blodgett films.

Key words: electric field, electret, silicon dioxide, water-repellency treatment

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Introduction. A stable electret effect, recently discovered in silicon dioxide (SiO_2) [2, 13], brings about the opportunity for using electrets based on this material as sources of constant fields of miniature electroacoustic transducers, since the manufacturing technology of the microelectret transducer can be combined with standard silicon micro technology [5, 6, 8]. Obviously, in this case it is convenient to use SiO_2 as an electret. Production of a stable electret based on this material is an urgent task, the use of such electrets will make it possible to widely introduce them into the modern standard planar silicon micro technology. This would allow the manufacturing of miniature electret microphones and other sensors built directly into the chip and manufactured simultaneously with the microchip in a single technological cycle. Although today there is considerable groundwork for controlling parameters, calculating and optimizing such structures [10-13], this task has not been finally solved because of the lack of stable in-time SiO_2-based electret. The problem of obtaining heavy rugged and stable electric charge (EC) in SiO_2 is of interest from the standpoint of the possibility of increasing the adhesion of various coatings deposited directly on SiO_2 or on other objects using the electret generated electric field. The strong influence of the electric field on adhesion has been proved both theoretically and experimentally [9, 14, 15, 17].

Of considerable interest is the use of SiO_2-based electrets in various technologies that are used or can be used in metallurgy and mineral processing. Let's consider a simple example. Before they can be used the large pieces of mined ore should be crushed. There are several types of breaking: primary, secondary, as well as fine grinding and powdering. Primary, secondary and fine breaking is carried out in crushing machines and powdering is done in mills. Breaking can be performed by crushing, abrasion, splitting, impacting and a combination of the mentioned methods. In these processes, the particle sizes decrease from about 1000 to 1 mm. Obviously, the considered processes are accompanied by vibrations and many other physical effects, especially acoustic ones. Obtaining this information with the help of electret converters (accelerometers, microphones, threshold radiation dose and dustiness sensors, etc.) is of great interest. It should be noted that electrified silica, since it is an inorganic material, significantly benefits in terms of its use versus traditional electrets based on polymeric materials. In addition, the possibility of miniaturization of sensors based on SiO_2 gives additional possibilities. Another aspect related to the

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prospects of using electrets in metallurgy and mineral processing is the external electric field of the electret. This field is similar to the traditionally used electrostatic field created by an external high voltage source, which has found application in various filters, separators, dust collectors, etc. The use of electrets offers the challenge for a significant reduction in the used electrical voltage or even refusal of it. As compared to polymers high hardness of SiO_2 favorably distinguishes this material from the traditionally used electrets.

Finally, the electrical field of SiO_2-based electrets may prove to be in demand for a wide variety of applications. It is known about its influence on biological objects and combustion processes, etc. [7].

In the first designs of semiconductor converters, a polymer film was used as the electret, which was attached on the surface of SiO_2. During assembly process they revealed significant deficiencies of such structures. The operations associated with applying the film to silicon dioxide are poorly consistent with the standard integrated technology. In addition, the film as attached at a high temperature using a plasma discharge, which worsens the electret properties of the fluoroplastic and prevents the serial production of such converters. The use of SiO_2 as an electret material allows to get rid of these drawbacks, while preserving the mentioned advantages.

Research methodology. Even though SiO_2 is widely used in semiconductor technology, many physical processes and phenomena that determine the performance characteristics of silicon dioxide, such as the ability to store embedded EC, the localization of this charge and the effect of surface phenomena on the decline of the surface potential are unclear. In this regard, intensive research is being conducted to study the mechanisms of EC appearance and localization in SiO_2, depending on the technology of its formation, as well as the possibilities of using non-destructive testing of the corresponding MIS structures.

At present based on the data of thermoactivation current spectroscopy [1, 2], it is considered that there are four types of traps and corresponding ECs in silicon dioxide:

1. EC trapped in the Q_{it} oxide, which is a charge of electronic states that are localized at the Si-SiO_2 interface region.

2. Fixed EC of Q_f oxide located at the interface region or in its close proximity.

3. EC trapped in the Q_{ot} oxide. This charge occurs, for example, in case of X-ray irradiation of structures or injection of hot electrons into a dielectric. The corresponding traps are more or less evenly distributed over the thickness of the oxide layer.

4. EC of mobile Q_m ions is due to the presence of positively charged alkali metal ions Li^+ Na^+ K^+ in thermal silicon oxide, as well as H^+ ions distributed over the total oxide volume.

Of all these types of charge three types, Q_{it}, Q_f, Q_m, apparently, cannot cause a stable electret effect due to their mobility or location. It can be assumed that the charge that affects the stability of the electret effect is created by carriers of the Q_{ot} type, which during the injection process replace the neutral trap centers. Probably, the formation of the Q_{ot} charge is associated with the dissociation of water in SiO_2.

The capture mechanism can be represented as a chemical reaction. The first stage is the formation of hydroxyl groups:

$$Si - O - Si + H_20 \leftrightarrow 2\ SiOH.$$

The second stage is the hydrolysis of water molecules on the SiO_2 surface:

$$SiOH + H_20 \leftrightarrow SiO^- + H_30^+.$$

It is assumed that further the incoming electron is captured by a proton-donor center:

$$SiO^- + H_3O^+ + e \rightarrow SiO^- + H_2O + H.$$

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This electrochemical reaction leads to the formation of a stable negative charge with a low probability of the appearance of a neutral trap in this place, which is confirmed experimentally [2].

The decrease of the charge injected into the electret can occur due to the presence of volume or surface conductivity, as well as the screening of this charge by opposite charges from medium, leading to a substantial drop in the electret surface potential at high ambient humidity [3, 16]. Therefore, to increase the stability of the electret effect, it is necessary to perform water-repellency treatment of SiO_2 surface by applying thin (nanosized) hydrophobic coatings.

Electrets are characterized by strong dependence of stability on preparation and surface modification methods. As shown by the conducted studies, this observation can be fully attributed to SiO_2-based electrets. Now considerable progress has been achieved in solving the problem of obtaining a stable SiO_2-based electret. Probably the most promising hydrophobic coatings for SiO_2 are polyimide nanolayer compositions – Langmuir-Blodgett films [2, 6, 8]. The use of these coatings makes it possible to obtain not only a stable electret, but also to combine its production with a standard silicon process using high temperatures. In a number of these experiments we made measurements of electret surface potential and participated in the design of SiO_2-based subminiature transducers. However, the most detailed results were obtained during the work on havign a stable electret from SiO_2. Unlike Langmuir-Blodgett films, which require special equipment, the possibility of using more accessible materials and technologies was studied.

Results and discussion. Three groups of different samples were studied: non-hydrophobized, hydrophobized with vinyltriethoxysilane (VTES) and hydrophobized with hexamethyldisilazane (HMDS). As a result of experiments, it was discovered that the annealed sample has high sheet resistance in comparison with the modified samples [11]. Thus, the characteristic property of the SiO_2 surface is the significant dependence of the resistivity on the initial state of the hydrate coating of the sample, and not only on ambient humidity.

Figure 1 shows the possible state of the SiO_2 surface considering the adsorption processes occurring on the surface: physical adsorption and moisture chemisorption, which can reduce the stability of the electret potential.

When the water vapor interacts with the SiO_2 surface the following reaction occurs

$$Si – O – Si + H_20 \leftrightarrow 2SiOH.$$

Subsequently, polar hydroxyl groups attract water molecules:

$$SiOH + H_20 \leftrightarrow SiOH: OH_2.$$

In case of water-repellency treatment the proton conductivity is impeded by the motion of protons in the hydroxyl groups and the formation of a conductive monolayer of water on the SiO_2 surface. For example, during HMDS treatment the following reaction occurs (Fig.2):

$$2SiOH + (CH_3)_3\ SiNHSi(CH_3)_3 \rightarrow 2Si – O – Si\ (CH_3)_3 + NH_3.$$

This treatment reduces the amount of hydroxyl groups on the surface but does not reduce the number of internal hydroxyl groups.

High-temperature oxidation of silicon is one of the most common methods of obtaining SiO_2. This method is also used for the formation of silicon dioxide films for electrets. Oxidation conditions (temperature, oxidizer pressing, oxidation medium, etc.) determine the growth rate and the number of defects in the oxide.

Oxidation can occur in a «dry» oxygen medium as well as during processes involving water: wet oxidation, oxidation «in water vapor», or oxidation in «wet» oxygen.

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When oxidizing in «dry» oxygen the following reaction occurs:

$$SiO + O_2 \rightarrow SiO_2,$$

during processes including water we have this reaction:

$$Si + 2H_20 \rightarrow SiO_2 + 2H_2.$$

Studies have shown that silicon dioxide obtained in «wet» oxygen medium possesses the best electret properties. This is probably due to the large number of Si-OH groups on the surface of the oxide in the «wet» SiO_2, which increases the effectiveness of hydrophobic coatings during the modification of the SiO_2 surface.

It has been found that other methods of obtaining oxide, for example, electrochemical or plasmochemical, do not make it possible to get silica with good electret properties.

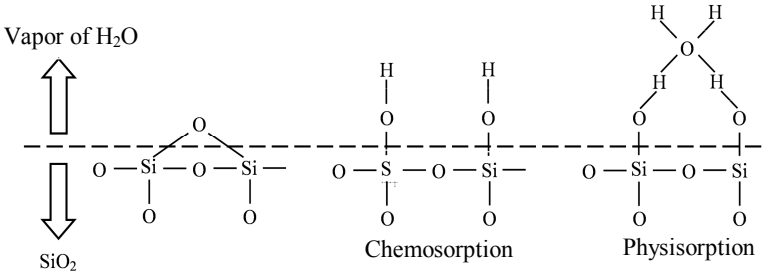


Fig.1. Water vapor sorption of the surface of SiO_2

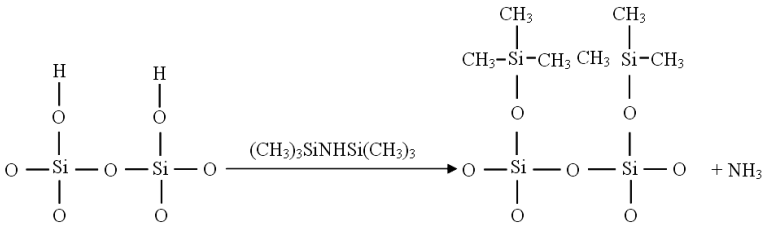


Fig.2. Reaction of formation of modified HMDS hydrophobic coating on the surface of SiO_2

As a result of the studies, batches of samples were found that showed a fairly good stability of electret surface potential U_e under room storage conditions. As can be seen from Table 1, the best stability results were shown by batches A and K (thermal oxide), D and E (plasmachemical oxide), and L (two-layer structure SiO_2-Si_3N_4). Conclusions were made about the advisability of using these samples in further studies, as well as the selection of appropriate technologies.

Table 1

Dependence of U_e decay on the storage time of charged sample under room conditions after charging

Batch	Sample no.	U_{grid}, V	U_{et}, V	U_e, V (after storage under room conditions)													
				2 h	1 day	2 days	3 days	5 days	6 days	8 days	9 days	12 days	13 days	15 days	19 days	27 days	29 days
A	A-1	–280	–270	–216	–222	–230	–214	–207	–209	–229	–220	–220	–	–221	–215	–	–220
	A-2		–260	–220	–214	–208	–234	–234	–228	–211	–215	–215	–	–220	–221	–	–218
B	B-1	–255	–166	–141	–135	–136	–128	–120	–123	–119	–119	–115	–	–113	–110	–	–109
	B-2		–166	–140	–126	–126	–125	–123	–121	–116	–116	–114	–	–114	–112	–	–110
C	C-1	–255	–251	–176	–157	–162	–163	–155	–148	–140	–131	–136	–	–135	–135	–	–132
	C-2		–250	–207	–170	–180	–176	–166	–164	–163	–170	–169	–	–158	–151	–	–149
D	D-1	–255	–257	–200	–204	–201	–203	–195	–187	–187	–187	–186	–	–175	–173	–	–173
	D-2		–257	–228	–227	–223	–221	–215	–202	–203	–206	–205	–	–202	–192	–	–189
E	E-1	–255	–256	–252	–252	–252	–250	–250	–253	–249	–245	–247	–	–247	–232	–	–232
	E-2		–256	–232	–208	–195	–197	–185	–185	–181	–174	–173	–	–173	–160	–	–153
F	F-1	–185	–81	–40	–24	–16	–16	–13	–15	–	–	–	–	–	–	–	–
	F-2		–105	–70	–28	–23	–17	–17	–17	–	–	–	–	–	–	–	–
H	H-1	–160	–69	–	–10	–	–4	0	–	–	–	–	–	–	–	–	–
K	K-1	–280	–280	–282	–282	–280	–280	–282	–280	–279	–273	–271	–	–271	–275	–	–280
	K-2		–280	–279	–283	–280	–279	–283	–285	–278	–274	–182	–	–278	–276	–	–276
L	L-1	–300	–298	–	–286	–	–279	–278	–276	–274	–274	–	–268	–	–	–	–263
	L-2		–296	–	–292	–	–290	–285	–285	–278	–278	–	–279	–	–	–	–266
M	M-1	–160	–93	–	–7	–	0	0	–	–	–	–	–	–	–	–	–
	M-2		–115	–	–18	–	–13	0	–	–	–	–	–	–	–	–	–
N	N-1	–160	–123	–	–29	–	–14	–19	–	–	–	–	–	–	–	–	–
	N-2		–122	–	–28	–	–19	–15	–	–	–	–	–	–	–	–	–

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Within the framework of this study we applied various types of hydrophobic coating that protect the film from accumulating moisture on the hydrophilic surface of SiO_2 and lead to a substantial decrease in the surface potential: HMDS (hexamethyldisilazane) and DMDCS (dimethyldichlorosilane).

Water repellents were applied to samples of K and D batches. D samples differ from each other in the mode of HMDS application process, which is expressed by the difference in the value of the wetting angle. After applying the hydrophobic coating, the most promising samples were tested in humid environment.

Samples were placed in a chamber with a relative humidity of 95-98% at $T = 20$ °C. The main test results are presented in Table 2.

Table 2

Dependency of U_e decay on time storage after sample charging under conditions of high humidity and after water-repellency treatment

Batch	Sample	Sample no.	U_{grid}, V	Wetting angle, degree	U_{et}, V	U_e,V							
						Sample storage time under high humidity conditions							
						1 day	3 days	4 days	6 days	10 days	13 days	17 days	27 days
1	K- DMDCS	K-1	–300	95	–303	–297	–292	–289	–287	–276	–283	–274	–270
2		K-2		95	–299	–297	–296	–297	–291	–294	–297	–280	–280
3	K- HMDS	K-3		73	–300	–300	–287	–285	–282	–278	–258	–252	–237
4		K-4		70	–298	–300	–295	–294	–290	–294	–293	–278	–270
5	D- HMDS	D-1		75	–298	–225	–147	–111	–74	–35	–26	0	0
6		D-2		80	–298	–290	–264	–260	–239	–207	–182	–150	–73
7		D-3		80	–298	–269	–260	–248	–220	–172	–137	–106	–34
8		D-4		77	–300	–192	–77	–61	–34	–21	–18	0	0
9		D-5		85	–295	–275	–268	–263	–257	–263	–267	–249	–205

It should be noted that for samples of one batch (in particular, D) treated with the same material, a correlation is observed between the increase in the wetting angle and the increase in the stability of the electret potential when samples are held in high humidity conditions, but for samples from different batches and treated with different hydrophobic materials this correlation is not observed.

We also tested the usage of positive high-temperature photoresist PTT-1-40 as a hydrophobic coating for SiO_2-based electret [4].

Hydrophobic material F-P, being a positive photoresist, is easily integrated into a standard planar process and is compatible with all standard etching, cleaning, and other procedures. This type of water repellent was applied to samples of batch A (the thickness of the silicon dioxide layer was 0.5 μm). The photoresist was applied by centrifugation. The thickness of the applied layer was about 0.4 μm. Then, the photoresist was exposed and samples of 10×12 mm^2 were formed. Charging was carried out in standard mode through a fluoroplastic shield with a window of 7×7 mm^2. The grid potential was 250 V. Then the charged samples were subjected to heat treatment at 200 °C for 1 hour. During this process the electric potential of the samples decreased as a result of the drift of the EC through the hydrophobic layer to the interface region of hydrophobic material and silicon dioxide. Further, three samples were tested under normal conditions and three more were tested under high humidity conditions. The results of testing the samples in room conditions and at high humidity are presented in Tables 3 and 4.

Table 3

Dependency of U_e decay on storage time under room conditions for samples treated with high-temperature hydrophobic material FPT-1-40

Sample	U_{grid}, V	U_{et}, V	U_e, after heat treatment, V	U_e, V					
				1 day	3 days	5 days	6 days	10 days	15 days
F-4	–250	–240	–169	–171	–170	–171	–171	–166	–166
F-5		–237	–161	–164	–164	–160	–162	–157	–159
F-6		–237	–171	–171	–168	–172	–170	–172	–171

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Table 4

Dependency of U_e decay on storage time under high humidity conditions for samples treated with high-temperature hydrophobic material FPT-1-40

Sample	U_{grid}, V	U_{et}, V	U_e, after heat treatment, V	U_e, V (humidity 95-98 %, T = 20 °C)					
				1 day	3 days	5 days	6 days	10 days	15 days
F-1	–250	–253	–143	–143	–133	–119	–118	–107	–95
F-2		–240	–142	–142	–135	–124	–120	–102	–76
F-3		–240	–144	–144	–140	–130	–125	–113	–89

The first experiment on the use of this material as hydrophobic coating gave the following results:

• heat treatment at 200 °C for 1 hour of electret led to approximately 30-40 % decrease in the potential, which, in our opinion, can be explained by the drift of the EC through the hydrophobic layer, followed by trapping the charge on the surface of silicon dioxide;

• when stored under room conditions for two weeks, the electret potential was practically unchanged;

• exposure to high relative humidity (95%) at room temperature for 10 days resulted in 28 % decrease in the electret potential, which is significantly worse than in the case of HMDS. However, there is a real prospect to significantly improve this result while continuing the experiments on working out the modes of application and use of the hydrophobic material.

On the basis of the conducted research it is possible to formulate the following variants of technological process for obtaining a stable electret from SiO_2:

The first option:

1. Thermal oxidation of the initial plate in the standard mode according to the TC technology (dry oxygen – wet – dry).

2. Heat treatment in vacuum with T = 550 °C, 1 h.

3. Application of HMDS from vapor stage. The plate should be cleaned and dried beforehand at T = 100 °C. Then it should be exposed to HMDS vapor heated up to T = 60 °C in enclosed volume for 10 min. The limiting wetting angle of samples treated with water-repellent should be not less than 80 degrees.

4. Charging of electret elements in the corona discharge using a three-electrode scheme. The required value of the electret element potential is set by the potential of the grid electrode and can vary from –50 to –300 V. The recommended charging time is 3 min.

5. Thermostabilization of the electret potential at T = 130 °C for 1 h.

The second option:

1. Thermal oxidation of the initial plate according to the TC technology (dry oxygen – wet – dry).

2. Heat treatment in vacuum with T = 550°C for 1 h.

3. Application of hydrophobic coating (photoresist F-P) with thickness of 0.1-0.4 μm by centrifugation.

4. Exposition for forming electret elements of a given geometry.

5. Etching of photoresist material.

6. Hardening (hardening temperature should be 20 °C above the maximum temperature used in subsequent technological operations). The maximum working temperature of the hydrophobic agent F-P is 450 °C.

7. Charging of electret elements in the corona discharge using a three-electrode scheme. The required value of the electret element potential is set by the potential of the grid electrode and can vary from –50 to –300 V. The recommended electrification time is 3 min.

8. Thermostabilization of the electret potential by heating at an average rate of 10 deg/min to T = 300 °C, followed by cooling at approximately the same rate.

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Conclusion. Based on the research results the following conclusions can be drawn:

1. To obtain a stable electret based on silicon dioxide, the oxide layer must be obtained in the oxidation regime in «wet» oxygen medium.

2. All studied hydrophobic materials have made it possible to substantially increase the stability of the SiO_2 charge to levels that allow such an electret to be used in some electronics devices.

3. The most promising water repellents are high-temperature photoresist FPT-1-40 and polyimide nanolayer compositions - Langmuir-Blodgett films.

4. The long-term stability of electrets based on silicon dioxide, required in electronics devices in several cases, was not achieved. This issue needs further research.

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