Structure growth, surface, and interfaces

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Investigation of nanosized structures using internal friction effect

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Abstract. The goal of this work is to study electrically active defects in planar (Si-SiO_2) and isoplanar (Si-SiO_2-Si_3N_4) silicon electret structures by the internal friction Q^{-1} method. The Q^{-1} set with a reversed pendulum type design is used for research. The activation energies and frequency factors of thermoelastic processes were determined due to the displacement of peaks on the Q^{-1} relaxation spectra. Moreover, additional local maxima formed after electrification of structures were found on the temperature dependence Q^{-1}. It is assumed that this may be due to the interaction of charged particles obtained as a result of irradiation in a corona discharge with capture centers, which are hydride Si-H and hydroxyl Si-OH groups, as well as with deep capture centers at the SiO_2-Si_3N_4 interface. We confirmed that the developed complex research method for determining the main electrophysical parameters of electret structures based on silicon oxide and silicon nitride allows finding optimal approaches to electrifying Si-SiO_2 and Si-SiO_2-Si_3N_4 structures for their practical application as active elements of electret sensors and actuators.

Keywords: Internal friction, Young's Modulus, electrets, silicon oxide, silicon nitride

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Материалы конференции
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Исследование наноразмерных структур с использованием эффекта внутреннего трения

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Аннотация. Целью данной работы является исследование электрически активных дефектов в планарных (Si-SiO_2) и изопланарных (Si-SiO_2-Si_3N_4) кремниевых электретных структурах методом внутреннего трения Q^{-1}. Для проведения исследований использовалась установка Q^{-1}, работающая по принципу обращенного маятника. Благодаря смещению пиков на релаксационных спектрах Q^{-1} были определены энергии активации и частотные факторы термоупругих процессов. Также, на температурной зависимости Q^{-1} были обнаружены дополнительные локальные максимумы, образующиеся после электризации структур. Предполагается, что это может быть связано с взаимодействием заряженных частиц, полученных в результате облучения в коронном разряде, с центрами захвата, которыми являются гидридные Si-H и гидроксильные Si-OH группы,

а также с глубокими центрами захвата на интерфейсе SiO_2-Si_3N_4. Результаты работы показывают, что разработанный комплексный метод исследования для определения основных электрофизических параметров электретных структур на основе оксида и нитрида кремния позволяет определять оптимальные способы электризации структур Si-SiO_2 и Si-SiO_2-Si_3N_4 для их практического применения в качестве активных элементов электретных сенсоров и актюаторов.

Ключевые слова: внутреннее трение, модуль Юнга, электреты, оксид кремния, нитрид кремния

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Introduction

Silicon planar technology in production of microelectronics and microsystem technology allows to create miniature sensors and actuators for various purposes, including based on the electret effect (electret microphones, pressure sensors). Base materials obtained using planar and isoplanar technologies are of particular interest as electret materials in the designs of such devices; these are silicon dioxide SiO_2, silicon nitride Si_3N_4 and their two-layer composition SiO_2-Si_3N_4. There are many ways to obtain such structures, for example, by high-temperature thermal oxidation and nitration of silicon, gas-phase and plasma chemical deposition, ion implantation, and others. At the same time, the whole variety of electronic processes occurring in silicon-based semiconductor structures depends not only on the structure and defectiveness of silicon itself, its oxide and nitride, but also primarily on the Si-SiO_2 and SiO_2-Si_3N_4 interface [1].

The electret materials used in microsystem engineering must meet the following physical requirements. Firstly, it is the temporary stability of the electric field created by electrets, and secondly, the stability of the characteristics to environmental influences.

The miniaturization of modern devices leads to the need to modernize existing methods of control and nanodiagnostics of their parameters. Scanning probe microscopy is a vivid example of a technology allowing to obtain images not only in the traditional concepts of microscopy (roughness), but also as an analytical response to physical parameters distributed over the surface of the analyzed material. Such capabilities are implemented both by creating special techniques for controlling the probe, and by modifying the probe materials.

Among the new parameters that are planned to be transferred to the field of nano- and subnano measurements are the parameters of the so-called internal friction Q^{-1}. At the initial stage of the work, it is necessary to study the features of the nature of the measured phenomena. We selected the methods developed in the dissertation [2] as the basic methods. Internal friction Q^{-1} is the ability of materials to dissipate the energy of mechanical vibrations, converting it through various mechanisms into heat. The internal friction Q^{-1} is the inverse of the Q-factor, which is characterized by the ratio of the energy stored in the oscillatory system to the energy lost during the oscillation period [3, 4].

Let us explain the physical nature of the experimentally observed attenuation of free bending vibrations of the plate by the example of considering the thermoelastic effect. When bending a uniformly heated thin plate made of a material that expands under heating, the stretched sections of the sample cool down, and the compressed ones heat up. Thus, deformation causes a disturbance of thermal equilibrium. Temperature equalization is accompanied by an irreversible transition of elastic energy into thermal energy and is one of the reasons for the damping of vibrations. This process of restoring disturbed equilibrium is called relaxation. If several relaxation processes with different relaxation times τ_i occur simultaneously in a solid, then the totality of all relaxation times forms the so-called relaxation spectrum. At the same time, by changing the frequency of forced oscillations, a sequence of resonant peaks of internal friction Q^{-1} can be distinguished. For

the purposes of analyzing film nanomaterials and technological processes, designs of the reversed pendulum type are the most effective [5, 6]. In such measurement schemes, only the surface layers are subjected to deformation effects of plate samples, and thus the possibility of integrally obtaining information about the properties of the entire coating layer is realized.

The method of analyzing nanomaterials using the internal friction Q^{-1} effect has proven itself in many applications. Among them, determination of the intensity of relaxation internal friction and activation energy in semiconductor compounds before and after electron irradiation [7]. Thus, the aim of the work is to study electrically active defects at the interface of dielectric films of silicon oxide and silicon nitride on a silicon substrate by the internal friction method.

Materials and Methods

The study of relaxation processes makes it possible to obtain information about the electronic structure of solids, electron-phonon interactions, the nature of phase transitions, the anharmonicity of interatomic interaction forces and the properties of various defects in semiconductor and dielectric materials. For Si-SiO_2 and Si-SiO_2-Si_3N_4 electret structures, understanding the nature of the relaxation processes taking place in them makes it possible to predict the charge stability in these materials.

The materials studied were oxide films grown on the silicon surface, as well as composite structures of silicon oxide and nitride on the silicon surface.

Silicon dioxide was obtained by combined thermal oxidation in the dry–wet–dry oxidation mode at a temperature of 1050 °C, a layer of silicon nitride was obtained by chemical deposition from the gas phase in the form of a mixture of silane and ammonia at a temperature of 1000 °C: the samples were rectangular plates measuring 5×15 mm^2. The thicknesses of the SiO_2 and Si_3N_4 layers were ~ 0.7 μm and ~ 0.1 μm, respectively. The plates were divided into two parts, one of which was not charged, and the second was electrified in a corona discharge to a surface potential of 300 V.

The measurements were carried out on the internal friction Q^{-1} set using the reversed pendulum method, shown in Fig. 1 [5], in the temperature range of 20–430 °C and frequencies of 0.5–2 Hz. Relaxation times τ_i depend on temperature and frequency, so it is advisable to go into the infrasound region to work at 'comfortable' temperatures.

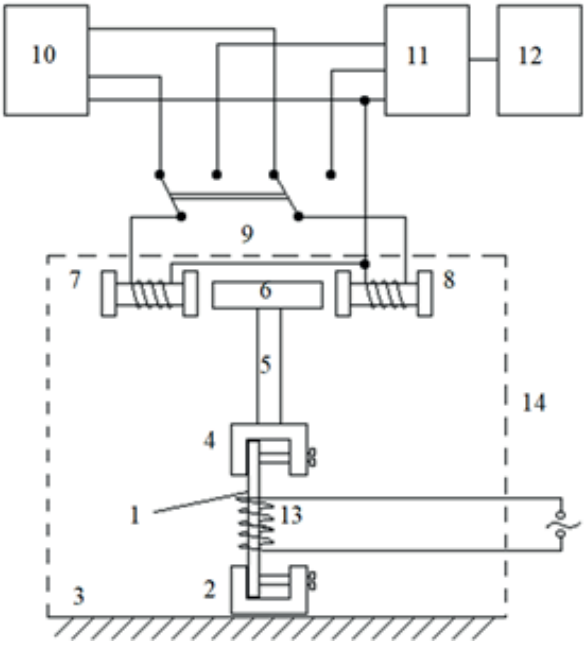

Fig. 1. Scheme of internal friction Q^{-1} set

The principle of operation of the Q^{-1} set is as follows. Sample *1* is fixed on edge using collet *2* to base *3*. Collet *4* with a pendulum *5* is attached to the other edge of sample *1*, in the upper part of which ring *6* of ferromagnetic material is installed. Near ring *6*, coils *7* and *8* are

symmetrically arranged, which, using switch 9, are connected either to low frequency generator *10* or to amplitude discriminator *11* connected to electronic counter *12* by an output. In the first case, coils *7* and *8* are used as the exciter of mechanical vibrations of the pendulum *5* due to the interaction of the magnetic field of the coils with ferromagnetic ring *6*, in the second case as a sensor for the movements of ring *6*. Heater *13* is located near sample *1*. Elements *1*, *2*, *4*–*8*, *13* are placed in airtight container *14*, from which air is pumped out to reduce the damping of the oscillations of pendulum *5*.

To determine the relaxation parameters (activation energy E_A and frequency factor ω_0) of the interphase boundaries, studies were carried out at different oscillation frequencies. This was carried out by changing the geometric sizes of the samples with automatic adjustment of the intrinsic oscillation frequency of the sample. When the frequency changed, the relaxation peaks shifted in temperature upward, which made it possible to determine the activation energies and frequency factors of thermoelastic processes ($E_{A1} = 0.28$ eV and $\omega_{01} = 3.2 \cdot 10^4$ s^{-1}, $E_{A2} = 0.39$ eV and $\omega_{01} = 1.0 \cdot 10^5$ s^{-1}).

The relaxation maxima associated with the excitation of the interface due to thermoelastic vibrations caused by the disordered structure of the Si-SiO_2 and Si-SiO_2-Si_3N_4 transition layers and the difference in the coefficients of thermal expansion were observed on the temperature dependence of the Q^{-1} of electrified samples.

With various methods of producing films, depending on the technological conditions in the structures under study, both tensile and compressive elastic mechanical stresses may be present. A more complex case of joint (and equivalent in magnitude) action of both (tensile and compressive) stresses is relatively rare. The value of elastic stresses is ~ 1–$5 \cdot 10^9$ dyn/cm^2 (on average $\sim 3 \cdot 10^9$ dyn/cm^2). One of the causes of elastic stresses is the difference in the coefficients of thermal expansion Si, SiO_2, Si_3N_4 [8, 9]. Elastic stresses in Si-SiO_2 and Si-SiO_2-Si_3N_4 structures depend on technological conditions: temperature and film deposition rate, characteristics of the annealing process, concentration of impurity defects and porosity of the film.

Results and Discussion

Common to all the uncharged samples studied was the detection of relaxation maxima on the temperature dependence Q^{-1} at a temperature of 180 °C for Si-SiO_2 and 100 °C and ~ 180 °C for Si-SiO_2-Si_3N_4 (Fig. 2).

Additional relaxation peaks were observed on the temperature dependence of the internal friction of electrified samples, which were absent in uncharged structures. Since the internal friction spectra of the studied samples did not change from tempering, it can be assumed that the appearance of an additional peak is due to the action of a charge injected into the sample volume using a corona discharge.

For electrified samples, this technique is informative enough to determine the energy position of the Q^{-1} peak associated with relaxation processes. The most interesting physical result is the ability to track changes in the temperature of the peak maximum, accompanied by a charge drain. Fig. 2 shows the Q^{-1} spectra for charged samples. With each subsequent measurement, the charge decreases, this is accompanied by the movement of the relaxation peak to the region of higher temperatures and, what is especially interesting, by an inflection and an increase in the elastic modulus in the peak region.

Within the framework of the model, it is assumed that during the electrification of a sample in a corona discharge, negatively charged ions are deposited on its free surface. As a result of electronic exchange with structural defects, excess electrons penetrate into the sample volume and are localized at the capture centers in the near-surface layer. Hydrogen-containing complexes, such as Si-H and Si-OH, as well as their own structural defects act as the main capture centers in layers of silicon dioxide and nitride [10]. Electron capture by Si-H and Si-OH groups occurs with electrochemical reactions and delocalization of atomic hydrogen. Under the action of an external or intrinsic electric field of the electret structure, charge carriers are released from the capture centers and flow deep into the sample until they are localized on the capture traps. In addition, charge carriers may be intercepted at deep capture centers concentrated at the SiO_2-Si_3N_4 phase interface.

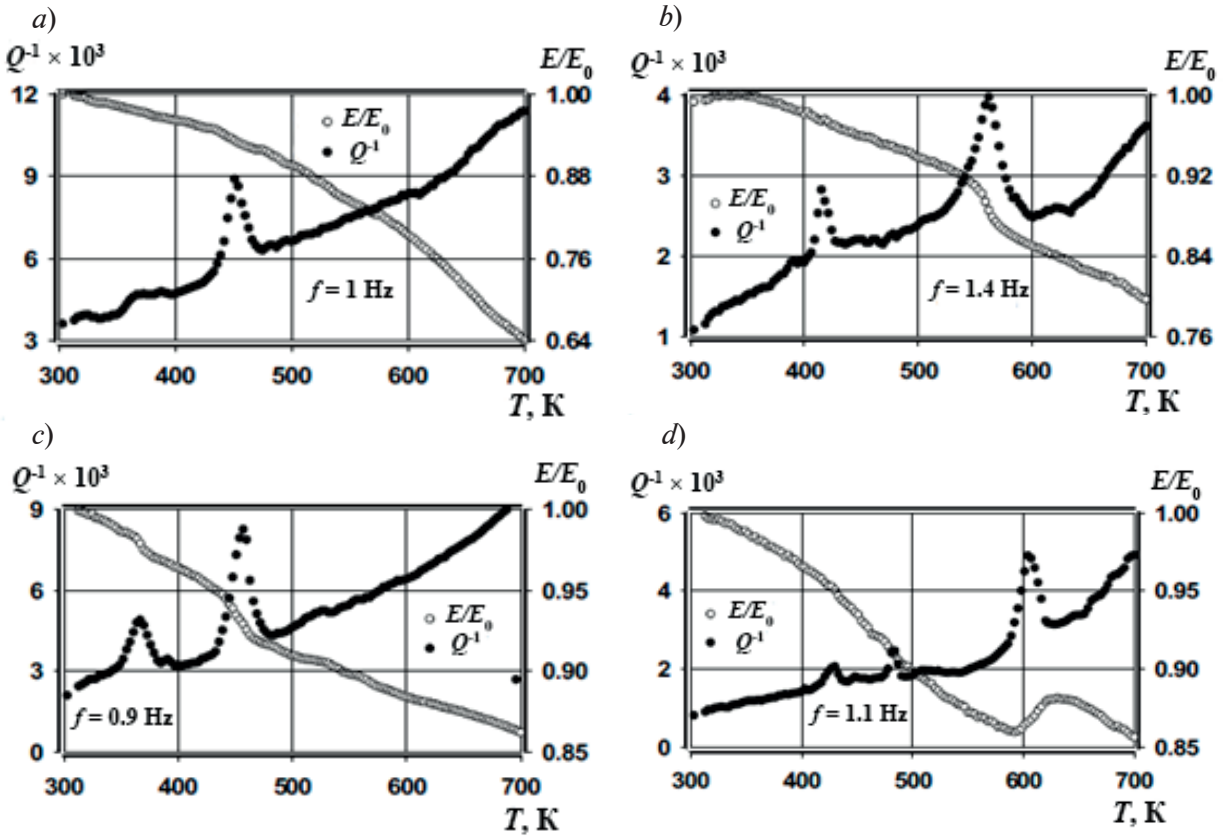

Fig. 2. Temperature dependence of Q^{-1} and change in Young's modulus of uncharged Si-SiO_2 (*a*), Si-SiO_2-Si_3N_4 (*c*) samples and negatively charged Si-SiO_2 (*b*), Si-SiO_2-Si_3N_4 (*d*) samples

Conclusion

It is shown that additional peaks associated with the excitation of new defects appear on the temperature dependence Q^{-1} of charged samples, which are absent in uncharged structures. This indicates that the charge carriers injected into the volume have a sufficiently strong effect on the defects in the structure of the materials under study. It is assumed that atomic hydrogen, delocalized during electrification during electron capture and decomposition of the impurity capture center, is responsible for these peaks.

A new method was proposed for studying relaxation processes in electrified samples, in which the dynamics of changes in the internal friction and Young's modulus of charged samples during sequential heating-cooling cycles was tracked. Under the influence of temperature, the charge carriers are activated, shifted deep into the sample and re-localized at the capture centers, thereby releasing more and more hydrogen.

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