INFLUENCE OF RADIATION EXPOSURE ON THE PROPERTIES OF DIELECTRIC LAYERS BASED ON ANODIC ALUMINUM OXIDE

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Abstract. Devices that are used in the aerospace industry must operate in extreme conditions, so it is important to understand how the properties of materials change under the influence of radiation and low temperatures. Anodic aluminum oxide, due to its mechanical and dielectric properties, is widely used in electronic devices with a high degree of integration. Radiation exposure can lead to degradation of the electrophysical parameters of dielectric films and can also change their chemical composition. The methods for studying the effect of radiation exposure on the dielectric properties of films are shown in this article. The research has been carried out and the results of the influence of α-particles on the dielectric properties of a porous film of anodic aluminum oxide during the influence of low temperature are presented.

Keywords: anodic alumina, radiation exposure, porous film.

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


Introduction

Porous anodic alumina is a material with a unique structure. Aluminum oxide film is an ordered structure of hexagonal cells with a pore in the center of the cell. Film parameters such as pore diameter, cell size, oxide film thickness, and structure ordering can be controlled during formation by changing the parameters of the anodizing process [1]. Anodic aluminum oxide films can be used as dielectric bases for integrated circuits, matrices for the synthesis of nanoscale materials, membranes and active elements of sensors. Due to this, alumina is widely used in various fields such as biomedical, nanophotonics, microelectromechanical systems and aerospace industry.

Materials used in the aerospace industry are exposed to various types of radiation. Ionizing radiation, which causes significant changes in the properties of materials, include x-rays and γ-radiation, fluxes of electrons, protons, neutrons, as well as the nuclei of atoms of chemical elements. γ-radiation, when passing through a substance, interacts with electrons and atomic nuclei, as a result of which free electrons appear in the substance [2]. The flow of electrons (β-radiation), when exposed to a substance, is able to penetrate to a shallow depth. In this case, electrons lose energy for the excitation and ionization of atoms, for elastic collisions with atoms, for bremsstrahlung radiation, which affects deeper layers of the material. α-particles are nuclei of helium atoms...
with a double positive charge. Energy losses during movement in matter are mainly spent on the excitation and ionization of atoms. With a decrease in the energy of α-particles, elastic collisions with the atoms of the substance occur, which leads to the displacement of atoms in the lattice. Devices with a high degree of integration can be sensitive to natural radiation even at the surface of the Earth.

Radiation can affect electronic equipment, degrading performance and resulting in data loss. Degradation of characteristics occurs due to the formation of electron-hole pairs in the gate and insulating dielectrics. Anodic aluminum oxide film shows good resistance to radiation [3] because it captures a significant number of electrons, which compensate for hole traps. In addition, Al₂O₃ has several levels of traps in the band gap, which facilitates easy tunneling of electrons from the dielectric to the substrate [4]. Exposure by medium energy hydrogen and helium ions can lead to the delamination of the anodic alumina film. Oxide delamination is caused by the residual stresses resulting from oxide growth and irregularities in the substrate [5]. The effect of radiation exposure on thin-film structures depends not only on the chemical composition of the films, but also on mechanical stresses at the interfaces [6]. In order to increase the radiation resistance of thin-film structures based on Al₂O₃, it is necessary to improve the technology of forming the interface between layers. To build devices operating in space, it is necessary to know what effect radiation exposure will have on the properties of anodic aluminum oxide films at cryogenic temperatures.

**Methods of conducting experiment**

Samples for research were made of A0H aluminum with a thickness of 0,9 mm. Substrates with the size of 60×48 mm were cut from a sheet of aluminum. Straightening was carried out to give the substrates flatness. The substrates were heated for an hour at the temperature of 400 °C to remove the internal mechanical stresses arising from the previous stages of processing. The removal of organic contaminants was carried out in a solution of potassium dichromate. The substrates were etched in a NaOH solution for 30 min to remove the upper deformed layer and improve the surface quality. For one-sided anodizing, a photoresist was applied to one of the sides of the substrate.

Anodizing was carried out in a specialized bath with a constant movement of the substrates and maintaining a constant temperature of the electrolyte. An oxalic acid-based solution was used as the electrolyte. Anodizing was carried out in a galvanostatic mode at a current density of 25 mA/cm². By varying the anodizing time, films of a given thickness can be obtained. After anodizing, the photoresist layer was removed.

Aluminum film was deposited to the surface of the anodic aluminum oxide to obtain a capacitor structure. The formation of the conducting layer was carried out in an electron-beam deposition unit. By varying the deposition time, it is possible to obtain an aluminum film of the required thickness. Substrates at different stages of manufacturing are shown in Fig. 1.

![Fig. 1. Substrates at different stages of manufacturing](image)

The samples were irradiated in a special device for studying the radiation properties of materials in a wide range of energies. The samples for the research were loaded into a special container made of a material that quickly looses its activity after irradiation. The container was placed in front of the water moderator. The distance from the water moderator to the sample is selected in
accordance with the required integral fluence of the particles. Particle fluence is controlled by placing activation foils next to the irradiated materials with subsequent measurement of the induced activity. The irradiation time depends on the required particle fluence and varies from several hours to 10 days. The temperature of the samples during irradiation does not exceed 50 °C.

Measurements of the electrical parameters of the capacitor structures in the temperature range from 4,2 to 320 K were carried out in a helium cryostat. For better heat transfer, the samples were attached to the massive copper base of the measuring cell using a thermally conductive adhesive. The change in the temperature regime on the sample during the measurements was carried out using an electric heater of the stove, mounted in the measuring cell. Temperature stabilization on the sample occurred 5 minutes after the change in the power released into the measuring cell. After that, the electrical capacitance of the tested samples was measured. The accuracy of temperature stabilization in the measuring cell was 0,2 K over the entire temperature range. The temperature was measured with a Cu-based thermocouple +0,15 at.%Fe paired with chromel. All contact wires coming from the sample were shielded. The measurement accuracy was no worse than 0,5 %.

Experimental results

The study of the effect of radiation on the dielectric properties of anodic aluminum oxide films was carried out using a capacitor structure in which the aluminum base was used as the lower plate and sprayed contact pad as the upper plate. 1 mm thick aluminum was used as a material for the test structures. Anodizing was carried out in a solution based on oxalic acid with constant stirring of the electrolyte in a galvanostatic mode. The electrolyte temperature was maintained at 15 °C. The thickness of the obtained anodic oxide films of aluminum was 60 μm. 1 μm thick aluminum film was deposited on the surface of the anodic aluminum oxide. Electric capacitance of the samples was measured using an R, L, C meter at a frequency of 1 MHz. The temperature dependence of alumina substrates was studied in a helium cryostat in the range from 4 to 300 K. After measuring the temperature dependence of the electrical capacitance of unirradiated samples, they were irradiated with α-particles with energy 5 MeV from source $^{239}$Pu with $3,1 \cdot 10^{14}$ cm$^{-2}$ dose. The measurement was repeated after irradiation. Temperature dependence electrical capacity of samples before and after irradiation is presented in Fig. 2.

![Fig. 2. Temperature dependence electrical capacity of samples before and after irradiation with α-particles](image)

Conclusion

Temperature dependence of the electrical capacitance for unirradiated and irradiated samples has the same form in the entire temperature range. This indicates the absence of any significant changes in the internal structure of the investigated dielectric layers. Quantitative differences in the capacity of unirradiated and irradiated samples are 2 %, the formation of radiation defects
in a dielectric leads to a decrease in its dielectric constant. Taking into account the high ionizing ability of α-particles and their high energy, it can be concluded that capacitor structures are resistant to this radiation effect.

References


Authors’ contribution

Biran S.A. developed an experimental technique, analyzed obtained data.
Korotkevich D.A. processed the obtained experimental data, prepared the manuscript of the article.
Korotkevich A.V. performed the task for the study, prepared the manuscript of the article.
Garifov K.V. conducted the research and obtained experimental data.
Dashkevich A.D. made samples for the research.

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