Formation of metal-oxide film nanostructures based on anodic alumina with uniformly distributed nanoscale Ta inclusions and their electrical properties

K V Chernyakova¹, I A Vrublevsky¹, E N Muratova² and V A Moshnikov²

¹Belarusian State University of Informatics and Radioelectronics, 6 P. Brovki Street, Minsk, 220013, Belarus
²Saint Petersburg Electrotechnical University "LETI", ul. Prof. Popova 5, Saint Petersburg, 197376, Russia

E-mail: SokolovaEkNik@yandex.ru

Abstract. The paper presents the results of studies of the formation of film-metal oxide nanostructures with islet tantalum inclusions on the basis of anodizing processes of bilayer Ta-Al films. The surface resistivity and the temperature coefficient of resistance of such nanostructures were studied. The results obtained show that the proposed method allows us to copy completely the cellular-porous structure of anodic alumina and to form a nanostructured tantalum film of islet character with a dielectric phase in places of pores.

1. Introduction
At present, the matrix of anodic aluminum oxide (AAO) with a regular ordered nanoporous structure is widely used as a relatively inexpensive and well reproducible template for obtaining various nanostructures [1–4]. One example of such an application is an electrochemical method of embedding nanowires valve metals (Ta, Ti and Nb [4, 5]) into the oxide cell bases, for forming nanoscale column autoemitters. The technology of autoemission structures formation is based on processes of self-organization at the nanolevel. It is one of the most promising in terms of improving the reliability of devices and reducing their cost.

In this paper, we show a new possibility of using a matrix of nanoporous AAO to solve nanotechnology problems associated with the formation of metal oxide film nanostructures. The results of studies of the formation of film metal-oxide tantalum nanostructures using the anodization of two-layer Ta-Al films and their characteristics, such as the surface resistivity and the temperature coefficient of resistance (TCR), are presented.

2. Experiment
Tantalum and aluminum films 30 nm and 1500 μm thick, respectively, were deposited by electron beam evaporation in a vacuum in the Oratorio-9 electron-beam evaporation equipment with a planetary mechanism for rotating substrates. Sital brand ST-50-1 was used as a substrate for the deposition of films. The films of porous AAO were formed by electrochemical oxidation of aluminum in an electrolyte based on oxalic acid. Tantalum layer was electrochemically oxidized through the pores of the anodic alumina together with aluminum.
The formation of island tantalum film nanostructures was provided by local electrochemical oxidation of the Ta layer through a mask of nanoporous alumina with open pores (figure 1). The use of such a pattern with a nano-sized pattern solved the problem of the formation of an ordered regular-distributed array of nanosized dielectric islands from tantalum oxide, leading to a disruption of the continuity of the tantalum layer in the resistive layer.

![Diagram](image)

**Figure 1.** Schematic representation of the formation of island tantalum film nanostructures.

The proposed process allows us to copy completely the cellular-porous structure of the AAO and to form a nanostructured tantalum island-like film with dielectric inclusions at the places of pores.

### 3. Results and discussion

Structured nanoporous films of aluminum oxide were obtained as a result of electrochemical oxidation of aluminum (figure 2).

![SEM images](image)

(a) (b)

**Figure 2.** SEM image of the morphology of a porous AOA film: surface (a) and profile (b).

The pore diameters in the obtained AOA films, and hence the sizes of the regions of the dielectric phase of the anodic tantalum oxide (ATO) formed in the tantalum film, range from 32 nm to 60 nm with distances between such regions from 92 nm to 136 nm. These parameters depend on the chosen anodizing voltage in oxalic acid (50–80 V).
In this work, the influence of various types of pore-forming electrolytes on the nature of electrical resistance and TCS of formed island tantalum nanostructures was studied. Figure 3 shows the temperature dependence of the change in conductivity for tantalum structures formed in an electrolyte based on oxalic acid at different anodizing voltage.

![Figure 3](image)

**Figure 3.** Temperature dependencies of the change in the conductivity of tantalum island nanosized films formed by two-layer anodization of Al-Ta films in 4% oxalic acid at voltages of 50, 60, 70, and 80 V.

It should be noted that, despite the metallic nature of the conducting substrate, the island films had a negative temperature coefficient of resistance. This indicates the presence of current transfer between isolated metal islands. In the case of the formation of film metal-oxide nanoscale structures, the effect of grain boundaries becomes an important factor. An increase in the dimensions of the dielectric fractions in the film leads to an increase in the resistance and to a further transition to the dielectric properties.

### 4. Conclusions

It was found that the formation of sections of the dielectric phase from an anodic tantalum oxide in a tantalum film and the influence of grain boundaries for tantalum inclusions in the metal oxide structure were the main factors that provided an increase in resistance and negative TCR of tantalum nanoscale islet films formed by two-layer porous anodizing of films Al-Ta.

### Acknowledgments

This work was supported by RFBR research project №16-38-60110 mol_a_dk.

### References

[1] Luchinin V V and Tairov Yu M 2006 *Nanotechnology: physics, processes, diagnostics, devices* (Moscow: FIZMATLIT)

[2] Muratova E N, Matyushkin L B, Mosnikov V A, Chernyakova K V and Vrublevsky I A 2017 *Journal of Physics: Conference Series* **872** 012020.

[3] Muratova E N, Matyushkin L B, Mosnikov V A, Chernyakova K V and Vrublevsky I A 2018 *Inorganic materials* **54**(6) 593–6

[4] Tatarenko N I and Kravchenko V F 2006 *Auto emission nanostructures and devices based on them* (Moscow: FIZMAT)

[5] Mozalev A, Sakairi M and Takahashi H 2004 *J. Electrochem. Soc.* **151**(11) F257–68