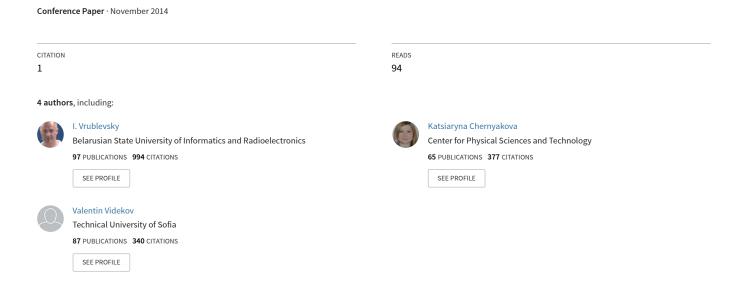
# Electromagnetic interference shielding characteristics of carbon-bearing porous anodic alumina



## BULGARIAN ACADEMY OF SCIENCES NATIONAL COORDINATION COUNCIL ON NANOTECHNOLOGIES

# NANOSCIENCE



NANOSTRUCTURED
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### ELECTROMAGNETIC INTERFERENCE SHIELDING CHARACTERISTICS OF CARBON-BEARING POROUS ANODIC ALUMINA

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**Abstract.** Electromagnetic interference shielding characteristics of carbon-bearing porous anodic alumina formed in a solution of oxalic acid were investigated in the frequency range of 8.2–12.4 GHz (*X*-band). Minimum value of reflective coefficient (–9.1 dB) and maximum value of EMR attenuation (3.7 dB) was reached after heating of the samples at 500 °C. Heating of the samples at temperatures of 600–800 °C results in decrease in EMR attenuation. The obtained data allow considering carbonbearing anodic alumina as promising materials for designing of composite shields of EMR.

**Keywords**: porous anodic alumina; amorphous carbon; electromagnetic shielding.

#### 1. Introduction

Porous anodic alumina has been demonstrated to be a material with wide range of electronic, mechanical, and adhesion properties [1]. So, it has considerable potential for designing of electromagnetic radiation (EMR) shields of new type. On the one hand, anodic alumina is insulator that possesses ordered porous structure [2]. On the other hand, samples obtained in organic acid solutions contain diamond-like amorphous carbon incorporated into alumina [3]; and amorphous carbon is known to be effective for EMR shielding [4]. It is also known that heat treatment affects the physical-chemical and optical properties of the carbon-bearing anodic alumina [5]. Therefore, the shielding characteristics of anodic alumina are likely to be changed by heat treatment. In the present study we aimed to reveal the effect of heat treatment on the shielding characteristics of carbon-bearing anodic alumina formed in solution of oxalic acid.

### 2. Experimental

The porous alumina was obtained by the doublesided anodizing of aluminum foil (99.99 %, 100 µm thickness, AlfaAesar) in 0.3 M solution of oxalic acid at a constant anodizing voltage of 50 V and temperature of  $(18.0 \pm 0.1)$  °C. Annealing of the samples was carried out at temperature range of 300-800 °C in air for 1 h in the programmable muffle oven Zhermack DM 50. The crosssection and surface morphology of the carbon-bearing anodic alumina were studied by scanning electron microscope JOEL 840A equipped with an energy-dispersive Xray (EDX) system. The scanning electron microscopy (SEM) results were processed by analysis program ImageJ. Electromagnetic interference shielding characteristics of the samples were investigated by panoramic voltage standing wave ratio (VSWR) and wave attenuation radar JA2R-67.

The reflective coefficient (R) was computed according to the following equation:

$$R = 20 \lg \frac{\text{VSWR} - 1}{\text{VSWR} + 1},\tag{1}$$

where

$$VSWR = \frac{1+\Gamma}{1-\Gamma},$$
(2)

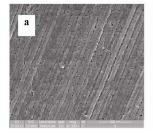
 $\Gamma$  is absolute magnitude of reflection coefficient:

$$\Gamma = \sqrt{\frac{P_r}{P_f}},\tag{3}$$

where  $P_r$  and  $P_f$  are powers of the forward and reflected waves, respectively.

### 3. Results and discussion

The anodic alumina membranes formed in solution of the oxalic acid have well-ordered porous structure with the following parameters: the pore diameter is about 42 nm and the interpore distance is about 102 nm (Figure 1, *a*). There are isolated, parallel cylindrical pores. The cylindrical pores are uniform and ordered in direction, perpendicular to the membrane surface (Figure 1, *b*). It is worth to note that due to the application of the double-sided anodizing technique the membranes were formed immediately, and aluminum was spent during oxidation process.



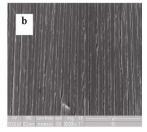


Figure 1. SEM image of the surface (a) and cross section (b) of carbon-bearing anodic alumina formed in the 0.3 M solution of oxalic acid at constant voltage of 50 V

The effect of annealing on the microstructure and pore geometry of the anodic alumina films was investigated in [6] by SEM. It was shown that the size and shape of the pores were not changed significantly up to 1000 °C. Therefore, the effect of pore size and shape on properties of the samples is negligible and will be not

taken into account in further discussions.

The composition of the surface of the "as-anodized" samples was studied by means of EDX spectroscopy. As it can be seen, there are Al-, O- and C-related peaks in the EDX spectrum (Figure 2). The quantitative composition of the carbon bearing anodic alumina is listed in Table.

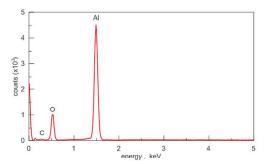


Figure 2. EDX spectrum of carbon-bearing anodic alumina formed in 0.3 M solution of oxalic acid at constant voltage of 50 V

Table 1

Element	Al	0	С
Content, at%	35.66	54.58	9.76
Weight, %	49.28	44.72	6.00

As it is known, anodic alumina formed in solutions of organic acids has a complex composition [7]. There are carbon-bearing components in the form of carbonates, carboxylates, carbonyls of aluminum, and amorphous carbon besides Al<sub>2</sub>O<sub>3</sub> and H<sub>2</sub>O [3]. The colour of the samples was not changed up to 600 °C and only after heating at 700 °C it turns black. At this temperature carbon-bearing components are not completely oxidized and carbonization process is likely to take place that results in the formation of black coloured carbon-rich clusters in the oxide bulkphase. Increase in annealing temperature up to 800 °C the samples become colourless that is caused by the oxidation of carbon-bearing components in the films. So, these results should be kept in mind while interpreting the spectra of reflection and transmission of EMR.

For "as-anodized" anodic alumina the reflective coefficient was about -7 dB. The heat treatment within the temperature range of 300–400 °C does not result in the significant changes in the spectra. After heat treatment at 500 °C the reflective coefficient decreases up to the -9.1 dB; and at 600–800 °C it increases up to -5.5–2.5 dB (Figure 3).

As it can be seen from Figure 4, the highest EMR attenuation was observed in the frequency range of 8.–9.5 GHz. It is characteristic for carbon-bearing materials. With increasing heating temperature from 300 to 400 °C the radiation attenuation does not change significantly and increases its maximum up to 3.7 dB at 500 °C. Heating of the samples at temperatures of 600–800 °C results in decrease in EMR attenuation down to 1.5–2.5 dB. The data obtained can be the evidence of oxidation of amorphous carbon in the anodic alumina during heat treatment in the air at temperatures above 600 °C.

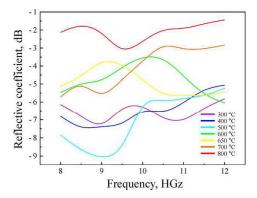
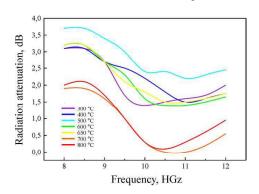


Figure 3. Reflective characteristics for carbon-bearing anodic alumina heat treated at different temperatures



**Figure 4.** Attenuation characteristics for carbon-bearing anodic alumina heat treated at different temperatures

### 4. Conclusions

Carbon-bearing anodic alumina formed in 0.3 M aqueous solution of oxalic acid has EMR attenuation of – 3.7 dB and reflective coefficient of –9–6 dB in the frequency range of 8.2–12.4 GHz (*X*-band). Minimum value of reflective coefficient (–9.1 dB) and maximum value of EMR attenuation (3.7 dB) was reached after heating of the samples at 500 °C. Heating of the samples at temperatures of 600–800 °C results in decrease in EMR attenuation. The obtained data allow considering carbon-bearing anodic alumina as promising materials for designing of composite shields of EMR.

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