Matrix metal oxide films formed on porous anodic alumina

G.G. Gorokh¹, A.I. Zakhlebayeva¹, A.I. Metla¹, V.V. Zhilinskiy², A.N. Murashkevich², N.V. Bogomazova²

¹Nanotechnology Laboratory, Belarusian State University of Informatics and Radioelectronics, Minsk220013, Belarus

²Department of Chemistry, Electrochemical Production Technology and Materials for Electronic Equipment, Belarusian state technological university, Minsk 220006, Belarus

Abstract. The metal oxide films of $Sn_xZn_yO_z$ and $Sn_xMo_yO_z$ systems deposited onto anodic alumina matrixes by chemical and ion layering from an aqueous solutions were characterized by scanning electron microscopy, Raman spectroscopy and electron probe X-ray microanalysis. The obtained matrix films had reproducible composition and structure and possessed certain morphological characteristics and properties.

1. Introduction

The semiconductor metal oxide films due to their high chemosensitive properties are among the most promising materials for gas sensors [1]. The formation of mixed metal oxide films and their structuring by applying of high ordered dielectric anodic alumina matrixes (AAM) allows to form the systems possessing a large active surface area and a heightened sensitivity and selectivity to various gases [2]. This paper presents the results of matrix-forming of metal oxide film by chemical sedimentation of transition metals into the porous anodic alumina as well as investigations of their morphology and microstructure.

2. Experimental

The initial specimens for deposition of metal oxide films were porous anodic alumina matrixes of 1 µm thickness and diameters of pores 120 nm, which were formed by two-step anodizing of the aluminium films on the silicon substrates [2].Matrix $Sn_xZn_yO_z$ systems were formed by chemical deposition of the metal oxide films from aquatic solutions on the AAM [3]. The chemical precipitation $Sn(OH)_2$ was carried out in 1% $SnSO_4$ solution with subsequent annealing to form SnO_2 films. The $Zn(OH)_2$ layers were prepared using aqueous solution of 0.01M $ZnSO_4$. The $Sn_xZn_yO_z$ system was formed by high-temperature annealing of deposited structures at $T = 750^{\circ}C$ for 30 min. Matrix $Sn_xMo_yO_z$ systems were formed by ion layering interleaved films of tin hydroxide from aqueous solution of $0.01M K_2[Sn(OH)_4]$ and molybdenum hydroxide from $0.01M (NH_4)_2MoO_3$ aqueous solution with following annealing at $T = 750^{\circ}C$ for 30 min. [4]. Such sequences of operations for the formation of $Sn_xZn_yO_z$ and $Sn_xMo_yO_z$ layers were repeated 10–30 times for a complete filling of the alumina templates pores.

3. Results and discussion

The surface morphology and cross-sections of the anodic films were examined in a Hitachi S-806 scanning electron microscope (SEM) operated at 15 kV of accelerated voltage. Fig. 1 shows SEM

images of the surface morphology (a,c) and cross-section (b,d) of the AAM with $Sn_xZn_yO_z$ (a,b) and $Sn_xMo_yO_z$ (c,d) films, from which we can see that the most uniform filling of matrixes is observed during the formation of structures by ion layering.



Figure 1. SEM microphotographs of the surface morphology (**a**,**c**) and cross-section (**b**,**d**) of the AAM with $Sn_xZn_yO_z$ (**a**,**b**) and $Sn_xMo_yO_z$ (**c**,**d**) films.

The composition and structure of the AAM with $Sn_xZn_yO_z$ and $Sn_xMo_yO_z$ films was clarified by Raman spectroscopy and the electron probe X-ray microanalysis. As an example, the Raman spectra of AAM/ $Sn_xZn_yO_z$ structures show the presence of crystalline phases with SnO_2 (550 cm⁻¹), Al₂O₃ (625 cm⁻¹) and ZnO (705 cm⁻¹, 820 cm⁻¹), which corresponded to the composition of metal oxide films deposited on the AAM (Fig. 2a). The electron probe X-ray microanalysis of $Sn_xMo_yO_z$ layers deposited onto the AAM demonstrated the presence of the following elements: Al (1,432 keV), O (0,56 keV), Si (1,77 keV), Mo (2,31 keV), and Sn (3,44 keV), and their quantitative ratio in the composition of the films (Fig. 2b).



Figure 2(a, b). (a) The Raman spectra of AAM/Sn_xZn_yO_z structures; **(b)** the energy dispersive X-ray spectrum of $Sn_xMo_yO_z$ layers deposited onto the AAM.

4. Conclusions

The developed technique based on the using of ordered AAM allows the functional films forming of different complex composition compounds with reproducible structure and properties for promising gas sensors and microsystems.

References

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