NANOSTRUCTURED MEMBRANES FOR MEMS BASED ON ANODIC ALUMINA

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The volumetric-surface variant of the capacitive MDM (metal-dielectric-metal) structure of the vertical direction based on high-ordered matrices of free anodic porous alumina membranes for applications in humidity sensing elements was designed. The improved humidity sensitivity was obtained due to the preparing of alumina membranes with open-ended and widened pores without a barrier layer. Such technological approach allows us to eliminate the effect of the electrolyte anions embedded in pore walls on adsorption and desorption processes in humidity sensing elements.

Nanostructured anodic porous alumina can be used as an active humidity sensing element in the humidity sensors [1,2] because the electrochemical process allows the capillary nanochannels to be formed and their geometrical parameters (diameter and length) to be varied. Anodic porous alumina membranes both with a dense alumina barrier layer at the pore bottoms and without this layer with open-ended pores can be used as starting material to design various humidity sensors. The structural parameters determine the sensitivity of nanoporous alumina to the humidity variation. These parameters are controlled by the electrolyte composition and electrical and temperature formation regimes. Conductive electrodes are possible to be formed either on one or both sides of the alumina membranes resulting in the fabrication of vertical or horizontal (interdigitated) capacitive relative humidity (RH) sensing structures.

The test sensing element designed for the humidity sensor based on the nanoporous alumina membrane is a volumetric-surface variant of the capacitive MDM (metal-dielectric-metal) structure of the vertical direction.

To improve humidity sensitivity, reduce response time and recovery time of the test sensing element designed, we use free membranes based on the high-ordered matrices of anodic nanoporous alumina with open-ended pores without the barrier layer.

Such membranes were formed by the two-stage electrochemical anodization in the 5% $H_2C_2O_4$ solution at the potentiostatic regime (45, 50, and 55 V) with the use of the barrier layer thinning method by slow voltage drop to 5 V at the final anodization stage combined with cathode polarization either in the 0.5M $H_2C_2O_4$ or in the neutral 0.5M KCl solution at (-4) V for 24, 27, 30, and 35 min for the alumina thicknesses of 40, 50, 60, and 70 μm correspondingly and with the alumina chemical etching in 5% H_3PO_4 for 5-45 min at 25-30°C. Such technology allows obtaining high uniformity of pore sizes and eliminating the effect of the electrolyte anions (O^{2-}, OH^-, and $C_2O_4^{2-}$) embedded in pore walls on the adsorption processes due to the decrease of embedded anion concentration at chemical etching.

Humidity permeable counter electrodes from the both sides of membranes formed by the metal (V, Ti, Ta, Al) film sputtering 50-200 nm in thickness were used as the conducting electrodes of the MDM structures. The metal film thickness was shown by the simulation to be not more than 3-4 d_p to provide alumina matrix with open-ended pores.

The study showed that the dependence of sensing element capacity on relative humidity (RH) at the RH increase from 10% to 90% and at the reverse RH decrease to the recovery of initial values and also a comparative analysis of the effect of the alumina structure parameters on the humidity sensor capacity at the RH variation.

According to the results the minimum values of the MDM nanostructure capacity are shown to be 22-35 pF at RH ~10% and amount to 370-390 pF at RH ~90%, i.e. the sensitivity of the humidity sensor is more than 4 pF per %. This indicates a high sensitivity index to allow signal digitizing at the electronic signal-conditioning circuit. Moreover, hysteresis value does not exceed 20 pF.

It was discovered that the comparative experimental values for the response (t_{res}) and recovery (t_{rec}) time during the adsorption process at RH increasing and the desorption process at RH decreasing for the sensing element based on the alumina free membrane with thickness of 50 μm and pore diameters of 70 nm. The observed recovery time is much shorter than response time. Kinetic testing procedures demonstrate that response time values are from 12 to 37 sec and recovery time data are from 3 to 8 sec correspondingly at RH increasing from 10% to 30, 50, 60, 70, 90% and RH decreasing from 30, 50, 60, 70, 90% to 10%. The explanation for such results might be that prepared membranes structures have the high-ordered matrix with open-ended and widened pores.

Thus, the improved humidity sensitivity, reduced response and recovery time over a wide humidity range were obtained due to the preparing and using of alumina membranes of thicknesses from 40 to 70 μm without the barrier layer with open-ended and widened pores from 51 to 89 nm in diameters. Such technology allows eliminating the effect of the electrolyte anions (O^{2-}, OH^-, and $C_2O_4^{2-}$) embedded in pore walls on the adsorption processes in humidity sensing elements due to the decrease of the embedded anion concentration at chemical etching and improving the performance of humidity sensors.

References

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