Influence of dopants on the performance of humidity sensitive elements, prepared by deposition of TiO_2 via sol-gel method

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Abstract: *The present research focuses on comparative measurements of the influence of complex supplements on the sensitivity of humidity sensors. In order to optimize these samples, thin porous films of TiO_2 were deposited, after their synthesis by sol-gel method. Three different dopants were added to the primary mixture in order to improve the properties of the films, expressed in water uptake capability. The latter is significantly changed either by increase in the film porousity, or by a change in its nature, shifting it to more hydrophilic.*

Key words: *Sol-gel method, sensitive elements, dopants, comparative measurements*

INTRODUCTION

The quality of regulation of each system is based on four basic parameters: 1- acceptable deviation; 2- the highest dynamic deviation; 3- retention time; 4- sensitivity and 5- range of measured values [1]. These output parameters depend on its labour principle of action and construction. The latter is completely predetermined by the method and the conditions used for the synthesis of the materials which compose the sensor, and even its design.

On the other hand, the sol-gel method has evinced as a versatile method for obtaining various products [2]. All sol-gel technologies could be divided into two general approaches: hydrolysis and polymerization of metal alcoxides, and dispersion of colloidal particles in liquid medium [3].

The general purpose of the present research is to compare the influence of different additives to the sensitivity of sensitive elements expressed by change of their resistance with respect to the humidity of the surrounding environment.

EXPERIMENTAL

Four samples of ceramic substrates with deposited metallic conductive pathways (shown on fig. 1.) were treated with sol-gel solutions with different dopants.

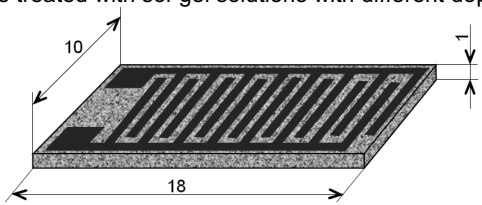

Fig. 1. General view of the samples

Description of the sol-gel solutions: the procedures were as follows: Liquid mixtures of terabutyloxititanate (TBOT) in C_4H_9OH, in ratio 1.5 to 1, were heated up-to 70˚C. The obtained solution was divided into three *preliminary solutions* (**PS**), with different additives, as follows: ***PS1-*** Bismuth acetate; ***PS2***-V^3-2,4 pentadionate; ***PS3***- Sodium tert-butoxide. While in the former two cases, the additions were preliminarily dissolved into the hydrolytic catalyst (HNO_3), in the last case the supplement was added directly to the solution. Thus, the obtained solutions were left at 70ºC for 30 min. These primary solutions were used to obtain *derivative mixtures* (**DM**), in order to investigate the complex influence of the additives.

They were obtained by mixing of the above described solutions (where every one was added in portion of 30ml), as it is described on table 1:

Table 1.

Order of mixing of the primary solutions (PS) to derivative mixtures (DM)

	PS1	PS2	PS3
DM1	+	+	----------
DM2	+	----------	+
DM3	----------	+	+
DM4	+	+	+

The obtained derivative mixtures were retained for 2 hours at 70 °C in covered beakers.

Deposition and annealing procedures: the obtained sol-gel solutions were deposited via "dip-coating" procedure, performed by triple dipping, subsequently: 20 minutes in solution, 20 minutes drying in desiccator. Afterwards, they were annealed at 400°C for 30 minutes, in order to obtain thin porous, chemically resistible ceramic superficial layer.

Measurements and characterization of the obtained samples: It was performed by Precision Impedance Analyzers 6505P product of Wayne Kerr Electronics Ltd, at 1kHz frequency and 500mV amplitude of the excitation signal.

It was connected to humidity conditioning chamber VAPORTRON H-100BL, produced by BUCK RESEARCH INSTRUMENTS L.L.C. It supplied precisely controlled humidity in range of 10 to 95% with maximal deviation up to 1.5% of relative humidity.

RESULTS AND DISCUSSION

After comparative systematic measurements, the following results were obtained, as they are shown on fig.2:

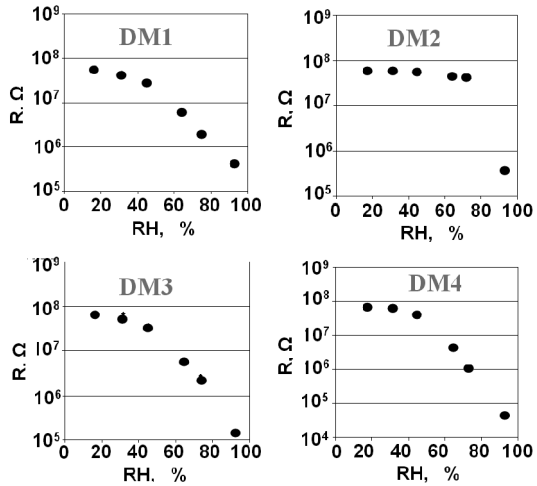

Fig. 2. Dependence between the electric resistance and the relative humidity

Fig 2 shows that the sample DW1, with superficial layer, prepared by addition of both of PS1 and PS2, reveals high level of sensitivity in the range of relative humidity, from 40% to 95%. In that range the resistance decreases with almost two orders of magnitude. The phase shift (which is indicator of the capacitive performance of the elements), changes

gradually with around 80°. That fact is evidence that the covering layer is remarkably capable to change its conductivity due to water adsorption.

The change of the resistance in respect to the relative humidity for the sample DM 2, prepared with both of SP1 and SP3, was insignificant in the range up to 70%. Its sensitivity reveals at higher values, between 70% and 95% of relative humidity. At that range the phase shift also changes with 80°.

The sample DM 3, prepared by addition of SP2 and SP3 shows remarkable sensitivity in relatively large range of relative humidity (from 30% to 95%). The resistance changes with two and half orders of magnitude. The phase shift also changes 80°.

The sample DM 4, which contains all of additions reveals remarkable sensitivity at the range between 40% and 95% of relative humidity, expressed by change of the resistance with three orders of magnitude. The phase shift also changes with more than 80°.

CONCLUSIONS AND FUTURE WORK

Taking in account the obtained results, the following conclusions could be done:

The addition of various supplements in the composition of the thin films influences significantly the sensitivity of the elements.

Each supplement has its individual influence over the range of the relative humidity, where the sensitivity of the elements reveals. Their effect occurs over the sensitivity, simultaneously with enlargement of the range of its revealing.

The most significant positive effect over both of increase of sensitivity and enlargement of the range of its expression belong to V^3-2,4 pentadionate & Sodium tert-butoxide;

Probably it changes the nature of the TiO_2, making it more hydrophilic. In that way the behaviour of the sensitive elements converts from capacitive, to resistive, with simultaneous decrease of the electric resistance.

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Докладът е рецензиран.