

ANODIC OXIDATION OF TANTALUM TO TANTALUM PENTOXIDE FOR HUMIDITY SENSORS

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1. Introduction

A capacitive humidity sensor includes a capacitor with two metallic electrodes. A moisture sensitive layer of tantalum pentoxide inserted between the electrodes serves as a dielectric. The dielectric is critical for humidity measurement in humidity sensors [1]. In common humidity sensors of this kind, polymer film is used as the dielectric layer. Some of humidity sensors are using oxides layer on a metal like aluminum, tantalum etc, or a combination of these layers, as a dielectric in a capacitor. The electrode based on Ta/Ta₂O₅ layers could be used as a gate in MOSFET under the interdigital structure [2]. According to the technology is final dielectric either homogeneous and nonporous (used only as a dielectric for separation of a capacitor), or inhomogeneous and porous by purpose. For creation of homogeneous is usually employed tantalum pentoxide layer prepared by anodic oxidation or by thermal oxidation. Another extreme is oxide layer, which is completely porous. In the case of introducing the porous layer to humidity, the water molecules are adsorbed, therefore is changing the capacity of the layer [1-3].

The process of anodic oxidation is explained with a following mechanism. On the surface of a metal electrode of humidity sensor is created a tantalum pentoxide layer after the tantalum electrode is dipped into the electrolyte. The intensity of an electric field in the Ta₂O₅ layer is very high, even up to 10⁷ V/cm. Under the influence of this field, oxygen ions migrate in the direction towards tantalum electrode and tantalum ions towards the electrolyte. This diffusion process take place until the electromotive force, created by the gradient of ions, is in the equilibrium with the outer voltage. After that is the oxidation process stopped. This method is used for very accurate and reproducible thickness of Ta₂O₅ layer. The thickness depends only on the applied voltage [2].

2. Experimental

For anodic oxidation of tantalum was designed and created a direct current voltage source. Maximum voltage is 48 V, maximum current is 25 mA. Optimal current density is 1 mA/cm² in galvanostatic mode. Anodic oxidation took place for 1 hour in the SIMAX beaker with different electrolytes. The temperature of the electrolyte was controlled. The set temperature was 25 °C with the deviation of 0.5 °C. The electrolyte was stirred during the process by electromagnetic stirrer for better distribution of the heat and stabilization of the solution's homogeneity. Negative electrode was created by an alumina plate with sputtered gold. Positive electrode was created by tantalum plate. Both electrodes (the alumina plate with gold and the tantalum plate) were before and after this deposition washed by water and dried for 1 hour at the temperature of 50 °C. The thickness of the layer after deposition was measured by Tencor Alphastep 200 Profilometer.

Electrolytes used for experiments were:

- a) Solution of ammonium citrate in water – the solution was prepared by dissolving 30 g in 1 l of distilled water and after a period of homogenization was filtrated and used for anodic oxidation.
- b) Solution of ammonium citrate in glycerol – the solution was composed from 1% neutral ammonium citrate in 90% glycerol solution (the rest – 10 % - was water). This solution was prepared by dissolving 12.43 g ammonium citrate in 121 ml of distilled water. After filtration was the solution completed with glycerol to 1 l.
- c) Solution of ammonium tartrate in glycerol – the solution was composed from 1% neutral ammonium tartrate in 90% glycerol solution (the rest – 10 % - was water). This solution was prepared by dissolving 12.43 g ammonium tartrate in 121 ml of distilled water. After filtration was the solution completed with glycerol to 1 l.

3. Results and Discussion

3.1 Anodic oxidation

The final color of deposited Ta₂O₅ varied according to the thickness of the layer. The film thickness of prepared samples was in the range between 100 and 600 nm. There was prepared a scale covering this range. The thickness was easily recognized by a different color.

The color range was from blue to grey. The typical thickness gained after 1 hour by the voltage of 48 V was deep violet with thickness around 200 nm.

The typical dependency of the voltage and current on the time are pictured in Fig. 1. After an initiation of the procedure (usually around 1 min), the anodic oxidation took place. There was no evidence of stating the equilibrium between the electromotive force and the outer voltage. For the film thickness was obtained an empirical formula:

$$d = 9 + 3.67 \cdot (U + 0.82) \quad (1)$$

where d is the thickness in nm and U is the applied voltage in V. Eq. 1 is valid only in the studied voltage range (from 10 to 48 V) and applied design of apparatus for anodic oxidation.

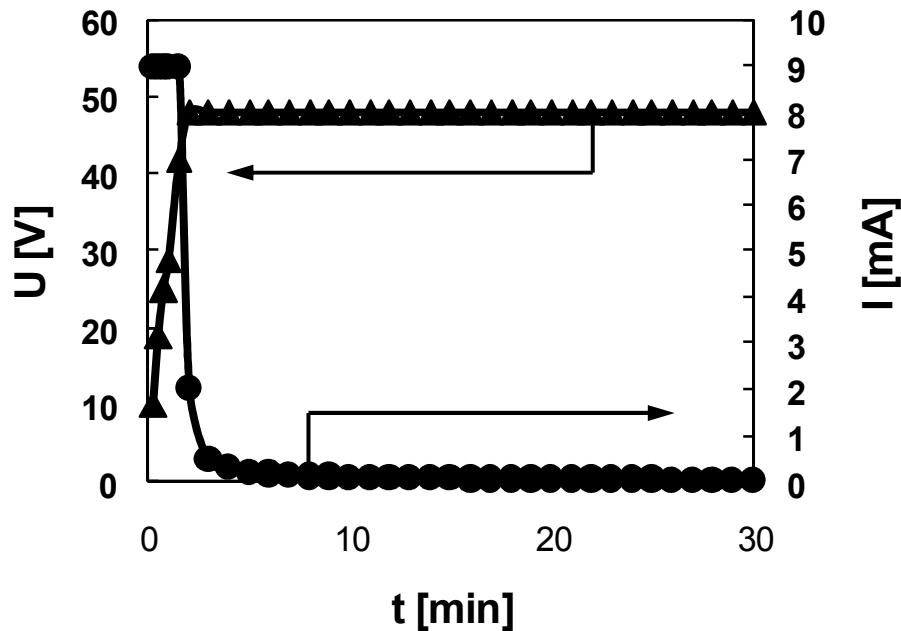


Fig. 1: Dependency of the voltage (▲) and current (●) on the time.

3.2 Tantalum pentoxide layer testing

As the main criteria for comparison of three tested electrolytes was taken the reaction on humidity. In the Fig. 2 are summarized the changes in capacity of the dielectric layer prepared by anodic oxidation in water solution of ammonia citrate. The reproducibility of the response is in relative scale good, but not in absolute. The next two electrolytes based on glycerol possessed almost the same results.

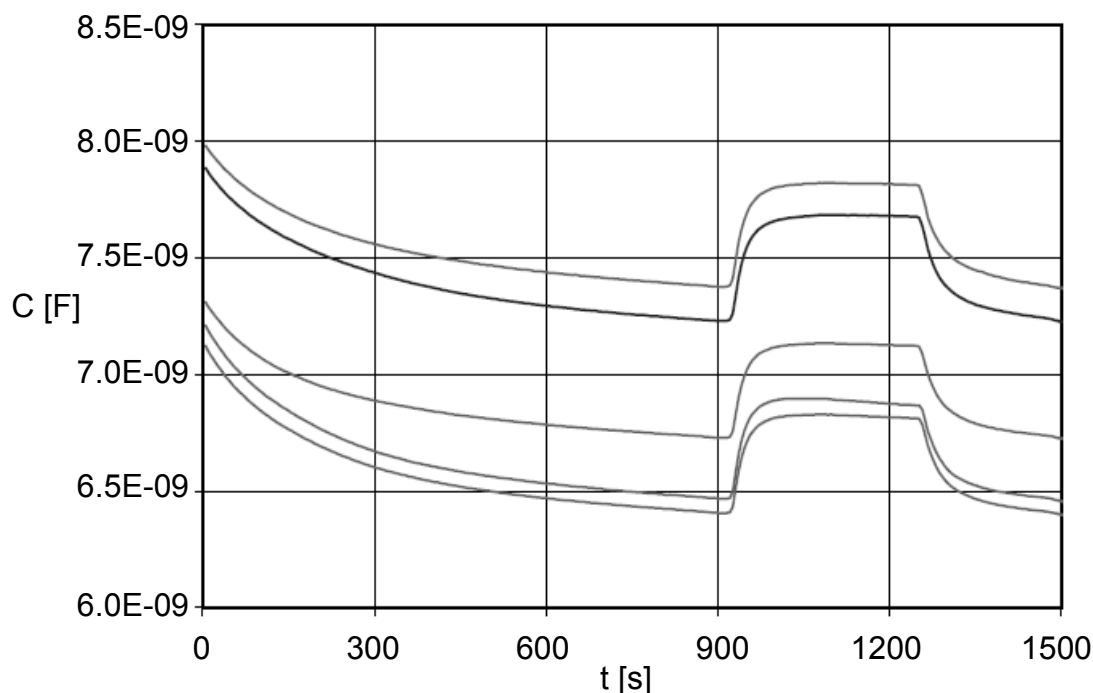


Fig. 2: The reaction on the humidity of 60 %. Different curves represent different samples.

4. Conclusions

In the comparison of three tested electrolytes wasn't best or worst one. Therefore the main outcomes of this work are optimized conditions for anodic oxidation of tantalum and an empirical description of tantalum pentoxide film thickness. The next step of our work will be optimizing the reproducibility of the layer properties.

Acknowledgement

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