SOME ASPECTS OF THICK FILM ELEKTROCHEMICAL SENSOR FABRICATION

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Miniature electrochemical sensors can be produced using thick film technology. Fabrication technology optimization of thick film sensors, the adjustment of optimal technological properties and especially the optimal properties of thick film electrode materials are main problems of sensor design. The thick film electrochemical sensors are usually made from commercial pastes which are originally developed for other applications. The producers' recommended firing temperature and time of firing for each paste are not optimal for electrochemical sensors. The output signal of electrochemical sensors increasing can be obtained by firing temperature and time varying. Some aspects about this problem are discussed in this paper.

Keywords: electrochemical sensor, technology optimization, firing temperature

1. INTRODUCTION

Fast expansion of information technologies and Internet brings a small interest of students to other electrotechnical and electronic branches and it also brings decreasing the number of experts in these branches. Successive introduction of secondary and high school pupils, university students and public is the most important condition for interest increasing. The area of microelectronic systems and technologies is one of the discussed branches. It is the main moving power of electronics and its development is very fast reflected in many other areas. An important part of microelectronic systems and technologies is an area of sensors and sensor systems, which hits almost into all branches of human activities. This is the main reason for accessing of this sensor technology area to students via easily available and comprehensible form e.g. multimedia educational system of Microsensors and Microelectromechanics systems [1]. This system is focused to microsensor, which are made semiconducting technology and think or thick film technology.

At the beginning the thick film technology (TFT) was focused on the production of hybrid integrated circuits [2]. The basic use of thick film technology was in the production of special integrated circuits, small series of nonstandard integrated circuits and prototypes [3]. At present time the TFT is partially suppressed in its classical meaning as a tool of preparation of very small electronic details by SMT. So the technological importance of TFT has been shifted significantly to military and high reliability applications and nonconventional applications. The nonconventional applications use the basic ideas of TFT, namely the screen printing as a method of active layer preparation and enhance the technology to printing of optically and chemically active layers. The examples of nonconventional applications include displays, heat spirals for pots, antennas for chip cards [4], fuses and especially sensor systems. Obviously an important area is open in nonconventional application for printing of materials which have chemical activity. This field includes TFT chemical sensors and biosensors [5], [6]. The main advantage lies in low price and small scale batch production. In many circumstances they are comparable with classical electrochemical cells with respect to sensitivity, linearity, while in many cases they have better mechanical and electrical properties.

The main goal of this work is the cognition and the theoretical description of the dependence of a TFT sensor response on the technological parameters. We will be focused on the case of amperometric TFT sensors only.

2. BASIC INFLUENCE

Processes on electrodes of TFT electrochemical sensors are substantially more complicated than in classical electrochemistry. The current output signal of amperometric sensor is depended on sensor design, materials, technological parameters of production, packaging, transport, storage and influences in measurement time (temperature, light, …).

The materials of electrodes are non-homogeneous from microscopic point of view (composition, structure...), they do not have well defined area, roughness of surface, etc. The examples of active electrode structure are on the fig. 1.

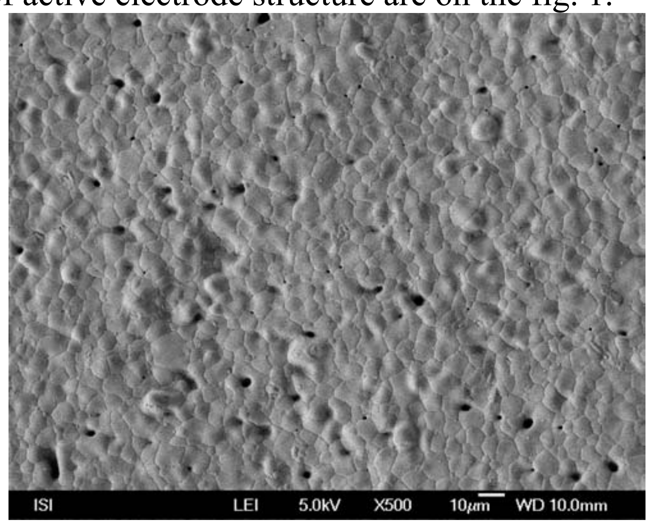

Fig. 1: The surface of active electrode structure.

Technology of TFT electrochemical sensors has grown from classical TFT materials. The majority of materials, which are routinely used for sensor preparation, are not designed for such applications. Electrochemical methods are extremely sensitive, which means that even small impurity which has not an influence in classical applications can influence the final sensitivity and other properties in case of the electrochemical sensor production.

Next the technical and technological parameters, which are recommended of paste producer, are optimal for standard application, but are not optimal for electrochemical sensor. The firing temperature is one of majority technological influences, which influences the sensor current output response. Study of this influence was done in this paper.

3. EXPERIMENT

3.1 Experiment objective

The study of the working electrode firing temperature influence to output current response of amperometric sensor.

Determination of optimal working electrode firing temperature of new amperometric sensor type using the standard detection of H_2O_2.

3.2 Chemicals

The practical measurements were done with model solution of hydrogen peroxide, which is sufficiently described in literature and is used very often in different branches of human activity (medicine, industry, cosmetics). The base solution concentration was 880 mmol l^{-1} H_2O_2. As a buffer solution was used 50 mmol l^{-1} $Na_2B_4O_7$ in H_2O.

3.3 Sample preparation

880 mmol l^{-1}, 88 mmol l^{-1} and 8,8 mmol l^{-1} hydrogen peroxide solutions in borate buffer were prepared as a testing solution. Hydrogen peroxide solutions were prepared just before the experiment, due to hydrogen peroxide instability.

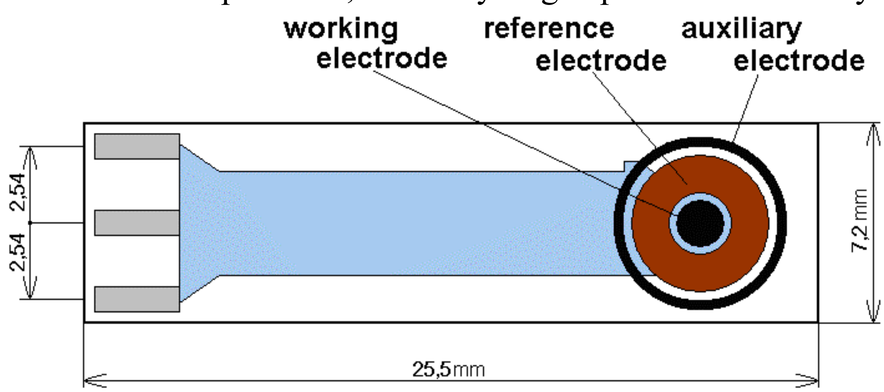

Fig. 2: Design of new amperometric sensor type.

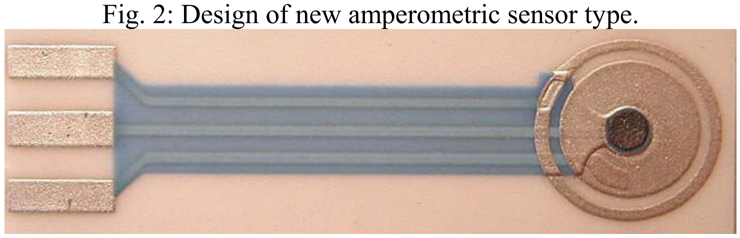

Fig. 3: Real sample of new amperometric sensor type.

3.4 New type of amperometric sensor

The new type of amperometric sensor was designed (fig. 2) and fabricated using standard TFT process (fig. 3). The TFT material used for Au working electrode was ESL 8881 B, for Pt working electrode was ESL 5545 and for reference and auxiliary electrode was ESL 9312 D. The working electrodes of amperometric sensor were fired at 820 °C, 835 °C, 850 °C, 855 °C, 865 °C and 880 °C. Firing time (40, 20, 10, 5 and 3 min) was reduced to velocity of transport band into a furnace (10, 20, 40, 80 and 120 mm min^{-1}).

3.5 Electrochemical experiment

The sensor was putted into a Micro Flow System [7] which was connected to an analytical electrochemical workstation AEW2 - 10 [8]. The amperometric method (on constant potential potential of 650 mV) was used for all measurements. Buffer solution volume was 10 ml. Addition of hydrogen peroxide was added four times from the stock solutions to measurement system for each concentration of H_2O_2. Addition volume was 50 μl. A calibration curve (fig. 4) was made for each sensor. Each calibration curve was interlaid by straight line whose slope was evaluated. The higher slope value of calibration curve is the better sensor quality is.

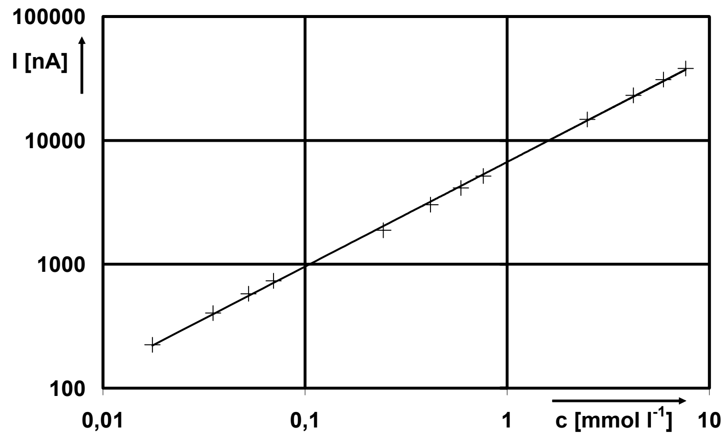

Fig. 4: Example of calibration curve of new amperometric sensor type.

3.6 Results and discussion

It can be seen that the slope of calibration curves is very dependent on working electrode firing temperature. The slope dependence of Au working electrode on firing temperature and velocity is shown in fig. 5 and fig. 6. Optimal firing temperature for standard application by producer is ϑ_{prod} = 850 °C. This temperature is not optimal in case of electrochemical application (slope is low). From the fig. 4 it is obvious that the optimal firing temperature for electrochemical applications is ϑ_{opt} = 820 °C (highest slope). The optimal firing velocity at firing temperature ϑ_{prod} = 850 °C is v_{opt} = 80 mm min^{-1} (firing time 5 min.).

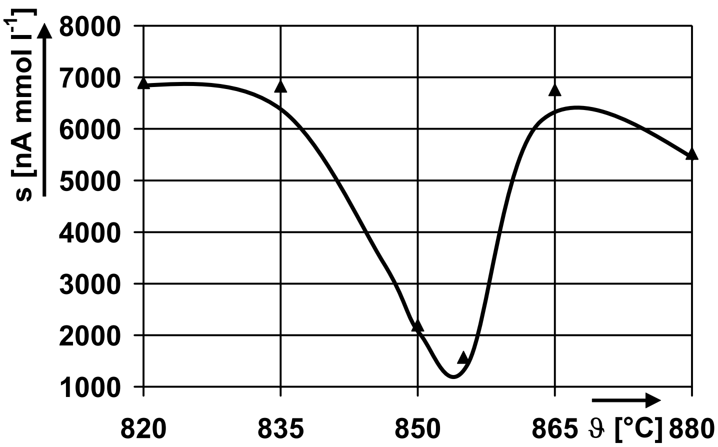

Fig. 5: The slope dependence of Au working electrode on firing temperature.

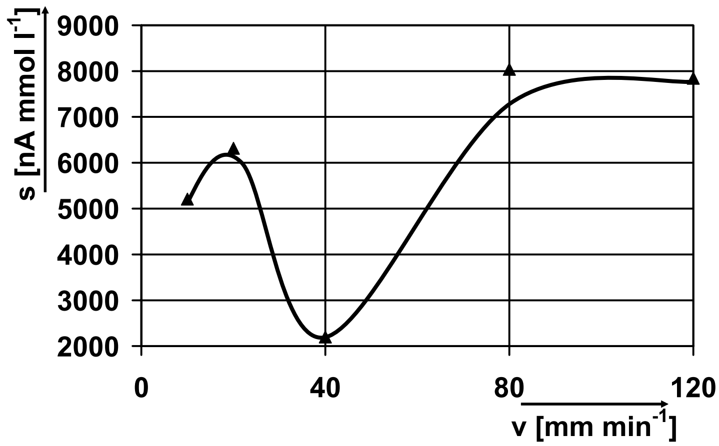

Fig. 6: The slope dependence of Au working electrode on firing velocity.

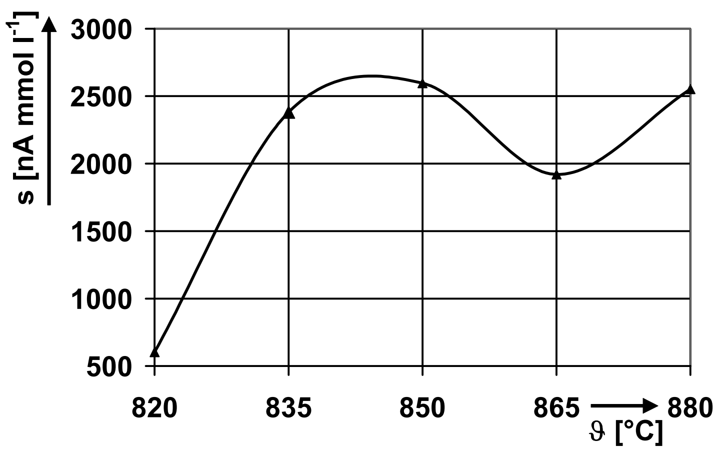

Fig. 7: The slope dependence of Pt working electrode on firing temperature.

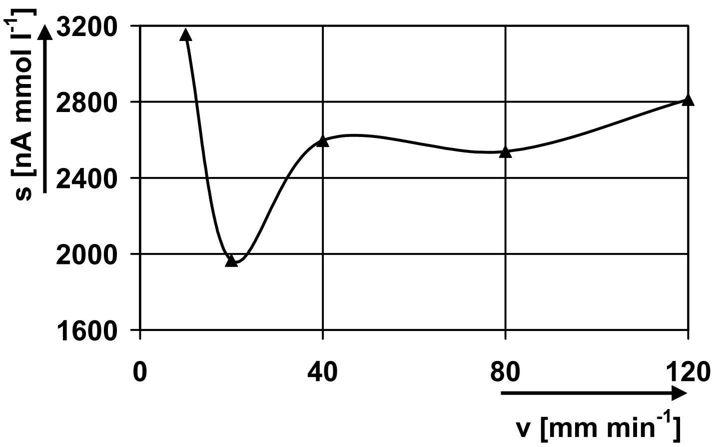

Fig. 8: The slope dependence of Pt working electrode on firing velocity.

In the fig. 7 there is shown the slope dependence of Pt working electrode on firing temperature and in the fig. 8 there is shown the slope dependence of Pt working electrode on firing velocity. The result shows that the optimal firing temperature is ϑ_{opt} = 850 °C. Optimal firing temperature for standard application recommend by paste producer is ϑ_{prod} = 980 °C and firing temperature range should be from 850 °C to 1300 °C. Recommend optimal firing temperature was not used, because sensor structure was destroyed in case of use of this temperature. The optimal firing velocity at firing temperature ϑ_{prod} = 850 °C is v_{opt} = 10 mm min^{-1} (firing time 40 min.).

4. CONCLUSION

The printing and firing are the basic operations in TFT production. In case of TFT electrochemical sensor production is firing process very important. TFT paste producer recommends an optimal firing temperature for standard applications only, but this temperature is not optimal for electrochemical applications. In this paper was determined the optimal firing temperature and optimal firing velocity for electrochemical amperometric sensor with Au (ESL 8881 B) and Pt (ESL 5545) working electrode.

5. ACKNOWLEDGEMENT

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