Nanostructured Microwire-based Micronanoelectrodes Intended for Biomedical Research

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Abstract — We explore the main advantages of a nanoelectrode application to biomedical research in comparison with a microelectrode application. Potential designs of glass-coated microwire-based nanoelectrodes are presented. The process of nanoelectrode tip shaping is studied. A relation between minimal nanoelectrode tip diameter and initial microwire nanostructure is demonstrated. A technological approach of nanoelectrode drawing process is proposed.

Index Terms — biomedical engineering, microwire, nanoelectrode, nanostructure, structural superplasticity.

1. BACKGROUND

Neuroengineering is a new, fast developing interdisciplinary science exploring fundamental mechanisms of signal transfer and potential to control reactions of central and peripheral nervous system. It comprises methods and achievements of clinical and experimental neurology, neurophysiology, biophysics, cybernetics, computer engineering, science of materials and nanotechnology [1].

One of the problems of brain function research is the measurement of potential of a neurocyte membrane. Micro- and nanoelectrodes of various designs and manufacturing methods are used in order to research neural network organization and other biological objects, [2-5]. In case of a mass-produced solid microelectrodes application for such purpose, a membrane encapsulation is damaged that does not ensure reliable long-term measurements.

Solid nanoelectrodes are not mass-produced, while the production of such nanoelectrodes in a laboratory is a complicated and labour-intensive technological procedure [6]. A promising material for the development of solid nanoelectrodes is a glass-coated cast microwire.

2. DESIGN OF SINGLE-CONTACT AND MULTI- CONTACT BIOMEDICAL ELECTRODES

The Institute ELIRI S. A. (Chisinau, the Republic of Moldova) has a long-standing experience in the development of glass-coated microwires and various microwire-based products. One of the lines of the Institute's activities is the development of micronanoelectrodes made of nanostructured metal- and alloy-based microwires.

We propose the design of a single-contact electrode which is shown at Fig. 1.

Allowable main overall dimensions of the electrode are listed below:

- a) minimal diameter of thinned metal core $(d) 200 \dots 1000$ nanometers;
- b) minimal diameter of thinned glass coating $(D) 1 \dots 5$ micrometers;
- c) length of thinned tip $(l) 4 \dots 10$ mm;
- d) angle of cone of thinning (α) from 2° to 6°;
- e) slendering angle (β) from 20° to 60°.

Besides single-contact electrodes, the Institute develops multi-contact ones as well as matrices of electrodes on the basis of filiform micro- and nanocompositions.

The design of a double-contact electrode as the first one in a series of multi-contact electrodes (with 2, 3 ... 7 contacts) is shown in Fig. 2.

The present type of electrode is intended for experiments with a bipolar stimulation, when a current injection must be limited by a relatively small area of a neural tissue, as well as for a simultaneous recording of activity of a neural substratum within a short distance between contacts of an electrode [3].

An allowable range of the main overall dimensions of multi-contact electrodes is shown below:

- a) minimal diameter of thinned metal core $(d) 1 \dots 5$ micrometer;
- b) minimal diameter of thinned glass coating $(D) 10 \dots 50$ micrometers;
- c) length of thinned tip $(I) 4 \dots 10$ mm;
- d) angle of cone of thinning (α) from 2° to 6°;
- e) gap between contacts $(t) 3 \dots 30$ micrometers;
- f) length of slendering $(l_1) 3 \dots 15$ micrometers;
- g) number of contacts (n) from 2 to 7.

The developed electrodes have the following advantages:

- geometric shapes and small overall dimensions allow recording of intracellular signals;
- smooth transition from glass-coating to metal core

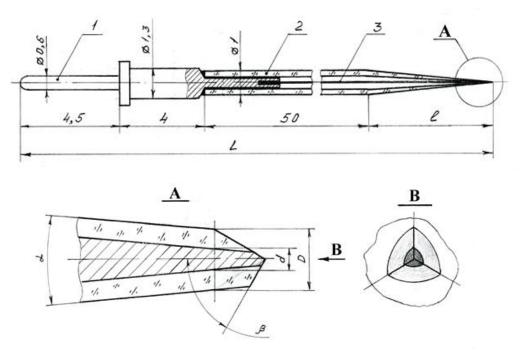


Fig. 1. Micronanoelectrode made on the basis of nanostructured glass-coated microwire: 1 - connector, 2 - glass capillary, 3 - microwire.

results in minimal tissue damage during electrode penetration;

- principle capability to produce electrodes with minimal diameter to 50 nanometers.

3. DISTINGUISHING FEATURES OF MICRONANOELECTRODE MANUFACTURING METHOD

The electrodes are manufactured by means of heating and drawing of a glass capillary along with either

a couple of microwire segments or a single one inserted into it with the help of a special equipment intended for pulling of micro-pipettes (a puller) type P-1000 produced by "SUTTER INSTRUMENT" (the USA) and further slendering of a thinned tip. A drawing procedure is carried out at temperatures below melting temperature of a microwire core.

An electrode tip shaping (a thinning procedure) during the said drawing process takes place as a result of an interaction of the following forces:

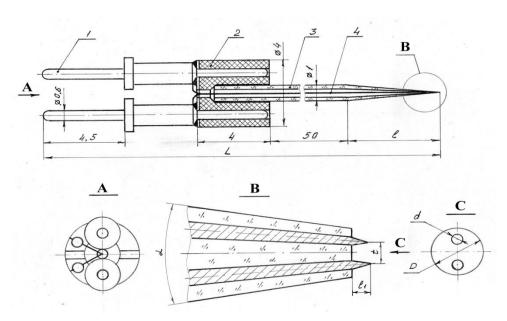


Figure 2. Double-contact micronanoelectrode: 1 - connector, 2 - body, 3 - glass capillary, 4 - microwire.

- 1) external axial tensile forces owing to a cohesion of a glass capillary with microwire insulation and microwire core with its glass coating;
- 2) an external radial pressing force of microwire core from insulation side and a glass capillary united with it (by this moment);
- 3) internal cohesion forces of granules (friction and attraction).

In a process of microwire core thinning its granules slip one relative to other and, finally, line up forming a filament consisting of maximal granules positioned one after another (in case of a given initial material). At the same time, gaps between granules can be filled in with amorphous phase of the material and smaller nanogranules. Thus, the minimal diameter of a thinned metal core is limited with maximal size of a nano-crystalline granule.

In the process of microwire manufacturing by means of Ulitovsky – Taylor method [7] the size of granules is defined by core material and its cooling rate. The latter significantly depends on main technological parameters of microwire casting process and first of all on microwire drawing rate, microbath temperature, wall thickness of a glass tube and glass tube feeding rate into an area of microwire shaping, cooling liquid temperature and speed, etc. [8]. Cooling rate increase results in reduction of granules size down to obtaining of amorphous structure [9].

Photos of one of tip areas of a single-contact electrode and 7-contact electrode on the basis of

ferromagnetic microwire demonstrating a visible nanostructured surface are shown at Fig. 3. Photos were taken by means of a light microscope type KH-3000V made by company "HIROX" (Japan).

In the process of microwire heating while making electrodes a re-crystallization of its initial structure can take place along with the generation of larger granules [10].

In order to detect the impact of the re-crystallization on a final granule size, the change of microwire core nanostructure has been studied prior and upon the electrode drawing procedure.

The studied microwire has been made at 250 meters per minute drawing rate with water cooling.

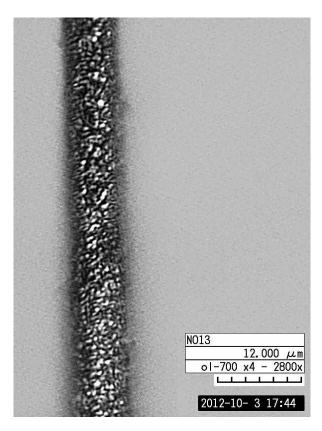
A picture of a 10x10 micrometers area of microwire core edge surface prior to a drawing procedure is shown at Fig. 4a.

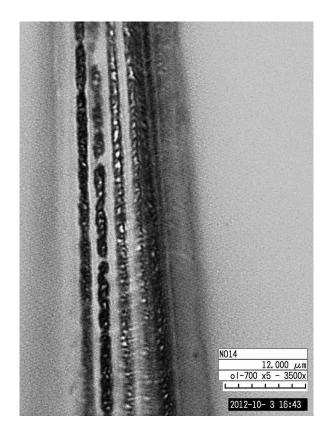
Granule sizes are from 200 nanometers to 500 nanometers. The picture has been taken by means of light microscope type KH-3000V with 3500X magnification.

A picture of a 10x10 micrometers area of side surface of electrode tip cone stripped from glass-coating is shown in Fig. 46.

Granule sizes of nanostructured surface are from 50 to 500 nanometers.

The image has been taken by means of a scanning probe microscope type NT-MDT NTEGRA Aura (Russia).





a) b)

Figure 3. Photos of a part of an electrode tip on the basis of a nanostructured ferromagnetic microwire: a) a single-contact electrode; 6) a 7-contact electrode.

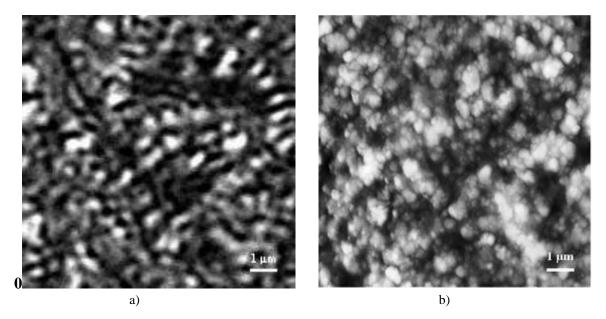


Figure 4. Photos of areas of a nanostructured ferromagnetic microwire: a) taken by means of a light microscope at 3500X magnification; б) taken by means of a scanning probe microscope.

The difference of visual minimal granule sizes at the said pictures is owing to different threshold resolutions of a light microscope and a probe one.

As one can see from these pictures, the morphology of a nanostructured surface and maximal granule size prior and upon a drawing process are actually the same.

The research has demonstrated that in case of such a short-term heating (from 1 second to 10 seconds) which take place in a microwre-based preform drawing process, actually, there is no metal re-crystallization.

Thus, minimal diameter of a thinned electrode tip is defined by nano-granule sizes of an initial microwire.

For further reduction of granule size, an increase of microwire drawing rate up to 1000 meters per minute and

higher is proposed, that will enable to achieve cooling rate about $10^7 \, \text{K/s}$.

In case of granule size less than 500 nanometers, its shape is close to a spherical one (so called spherulite), that results in a less cohesion between granules and, finally, in an increase of plastic properties of the material [11].

An electrode drawing process is divided into four steps:

1) glass capillary drawing along with a reduction of its inner diameter down to outer diameter of microwire coating with a minimal gap between them and shaping an initial electrode cone;



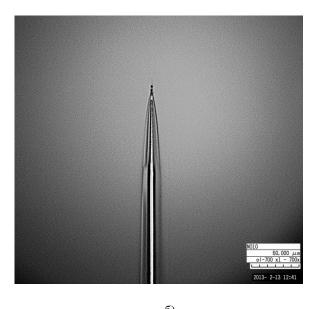


Figure 5. Images of drawn electrode segments: a) transition from an initial cone to a middle cone (a seam is visible in the center of the image); δ) transition from a middle cone to a tip.

- 2) joint drawing of a glass capillary and a microwire, as well as shaping of a seam between an internal capillary surface and an external microwire coating surface (generation of a united preform);
- 3) drawing of a united perform, i.e. a united part of an electrode and shaping its middle cone;
- 4) rapid drawing of a middle cone of an electrode perform and shaping its tip.

In order to obtain a required geometry of each part of an electrode, steps 1-3 may be split into several cycles each.

Each drawing cycle is carried out with the following settings and control of them:

- initial temperature inside of a drawing chamber;
 - heating temperature of drawing area;
 - basic drawing rate with heating off;
 - turning rapid drawing on or freezing it;
- delay time between cooling switching on and heating switching off;
 - · cooling duration;
 - · cooling air pressure;
 - hreshold of rapid drawing (force).

One of the versions of drawn electrode segments is shown in Figure 5.

The performed research has demonstrated the potential of production single-contact and multi-contact nanoelectrodes on the basis of nanostructured glass-coated microwires.

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