Multi-electrode silicon microprobes fabrication process for brain-computer interface

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Abstract. Multi-electrode microprobes fabrication process based on silicon substrate was developed using surface micromachining and anisotropic wet etching. The process flow consists of 20 main operations, including 4 lithography steps using 4 photomasks. The minimum size of the elements is 2 μm. The effect of the solution concentration (from 10 to 40% at 80°C) on the etching rate and surface roughness was studied. The optimal value of solution concentration leading to the formation of surface with the lowest root mean square roughness value was determined. The etching rates of monocrystalline silicon (100) face and silicon oxide were 1.5 μm/min and 10 nm/min, respectively. Rapid thermal annealing at 600 °C for 3 min increased the resistance of silicon oxide to the action of an alkaline solution by 2 times. As a result, the neural probe structure including two microprobes and electrical interface of 10 electrodes was fabricated.

1. Introduction
A study of brain activity and the development of neurocomputer interfaces is an actual scientific direction [1-4]. It is impossible without the development of new methods and devices. Neural probe is a highly informative instrument of brain activity study [1-3]. It is currently can be used for the formation of brain-computer interface and diagnosis of brain diseases in clinical settings [3]. The typical design includes a neural probe body, microprobes, and electrical interface. The electrical interface of the neural probe allows to record the electrical activity of many single neurons [1-3]. It consists of arrays of reading electrodes located on microbeams and connected by conducting lines to the contact pads on the body. One of the main problems in the use of neural probes is a damage of brain tissue during implantation of microprobes. It is proportional to the volume of the immersed part of the probe to the microlevel. Negative consequences are possible to decrease by reducing the overall dimensions of the probe to the microlevel. This can be achieved using bulk and surface micromachining [4-6].

There are important requirements for electrodes, such as biocompatibility and operational stability their parameters [2]. Therefore, they usually consist of several layers of metals, for example, chromium, titanium, silver, gold, and platinum [1-4]. The multilayer metallization with a total thickness of 300-400 nm forms by lift-off photolithography [6,7].

Microprobe materials should have enough margin of safety to resist the mechanical stresses that arise when the probe is inserted and removed. Silicon is the main structural material of microprobes despite the inherent brittleness [1-3]. Silicon probes can be inserted to a greater depth, in comparison with polymer structures. The minimum thickness determining the strength of the silicon microprobe structures is 20-50 μm. The plasma and thermal silicon oxide combination layers, as well as silicon
nitride can be used as an insulating and protective layers in neural probes [6]. The desire for a minimally invasive action and more precise positioning determines the need to improve neural probe concerning both its design and technological process.

The purpose of the work is to develop the fabrication process of a minimally invasive multi-electrode microprobes using surface micromachining and anisotropic wet etching of silicon.

2. Experimental details
The development of the fabrication process was carried out to implement the typical structure of a neural probe based on monocrystalline silicon. The neural probe structure includes a body with microprobes and a multichannel electrical interface isolated by a dielectric layer (figure 1).

![Figure 1. Structure of neural probe including (1) body with microprobes, (2) isolation layer, (3) electrical interface.](image)

The design and development of the fabrication process were carried out for the example of neural probes containing several (two or more) microprobes. It applies for the study of brain activity by a few-channel (from 2 to 16 channels) information processing system. There were adopted a few structural and technological limitations, such as the minimum element size of 1-2 μm, single-layer metallization, and ease of fabrication process. This determined that a surface micromachining and wet etching will be used to form the body and microprobes of the neural probe structure as well as lift-off photolithography to organize the electrical interface.

Several experiments were carried out to determine the optimal technological conditions for the manufacture of neural probes. The solution of potassium hydroxide (KOH) at a concentration of 10 to 40% for 80°C was used to anisotropic wet etching of monocrystalline silicon. The etching rate and surface roughness were controlled by stylus profilometry (AlphaStep D-100) and atomic force microscopy (NTEGRA Vita). The effect of rapid thermal annealing (STE RTA70) at a temperature of 600°C for 3 min on the mask resistance was studied. The SiOx/n-Si (100)/SiOx structures were used for this experiments.

The topology of layers on the front and back sides of the substrate was formed by photolithography (MJB4) using masks of silicon oxide and positive photoresists. The electrical interface was formed by lift-off lithography using LOR/Ultra i photoresists. The masking and isolation layers of silicon oxide were formed by plasma chemical vapor deposition (Plasmalab100) and rapid thermal annealing [7,8]. Silicon oxide was etched in buffer solution (HF (49%) : NH4F (40%) = 1:6) [9].

The set of photomasks with topological cells for the formation of pairs of counter-directed probes suspended on a frame was designed and used to implement a particular case of neural probe structures.

3. Results and discussion
The developed fabrication process based on 4 photolithography steps. The process flow includes following steps:

1. Chemical cleaning of a silicon substrate (RCA).
2. Producing of silicon oxide of thickness 200 nm at both sides substrate: isolation and protective layer (figure 2a).
3. 1st lithography (lift-off): electrical interface patterning.
4. Electron beam evaporation of metal system of thickness 400 nm.
5. Remove photoresist (lift-off) and metal in ultrasonic bath.
6. Plasma chemical deposition of silicon nitride of thickness 0.5 μm: protective layer.
7. Rapid thermal annealing.
8. 2nd lithography: silicon nitride layer patterning to the contact pads and electrodes, front side probe topology patterning.
9. Plasma chemical etching of silicon nitride and oxide [10,11] (figure 2b).
10. Anisotropic wet etching of monocrystalline silicon to a depth of 30-50 μm (figure 2c).
11. Plasma chemical deposition of silicon oxide of thickness 2 μm on the back side: protective layer.
12. Rapid thermal annealing.
13. 3rd lithography: back side probe topology patterning.
14. Plasma chemical etching of silicon oxide.
15. Plasma chemical deposition of silicon oxide of thickness 2 μm on the front side: protective layer.
16. Rapid thermal annealing.
17. Anisotropic wet etching of monocrystalline silicon on the back side (figure 2d).
18. 4th lithography: microprobe ends profiling by focused ion beam [12,13].
19. Wet etching of silicon oxide.
20. Mechanical separation of the neural probe from the frame.

![Figure 2](image_url)  
*Figure 2. Cross sectional view structure neural probe at various stages of the fabrication process.*

The effect of the solution concentration (from 10 to 40%) on the etching rate and surface roughness was studied. The etching rate of monocrystalline silicon at a fixed temperature varied nonlinearly from 1.1 to 1.5 μm/min for (100) face with increasing of the solution concentration. Wherein, the solution of potassium hydroxide of 27-30% was optimal and provided the smallest surface roughness of 13 nm.

The etching of silicon occurred irregularly on the surface substrate at the first experiments. It can be explained by the presence of some defects in the plasma films of silicon oxide and nitride. It was proposed to turn on the rapid thermal annealing of films to eliminate or at least reduce this effect. As a result, films became denser and uniform. The etching rate of silicon oxide decreased from 10 to 4.4 nm/min.

A series of neural probe structures with two microprobes was produced (figure 3). The thickness probe and midline width were 30-50 μm, 60-70 μm, respectively. The probe length was not more than 5 mm. The anisotropic nature of the silicon etching process determined the trapezoidal cross-sectional shape of the beams. The electrical interface of the neural probe was represented by 10 electrodes.
4. Conclusion
The fabrication process of microprobes for the study of brain activity was developed. The optimal technological conditions of anisotropic wet etching of n-type monocrystalline silicon are determined. The series of samples of neural probe structures with 10 electrodes was fabricated. The thickness and midline width of each probe were 30-50 μm and 60-70 μm respectively. The results of the work can be used for development and fabricating of multielectrode neural probe with a different number of microprobes.

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