Monitoring of the Bioconversion Processes

Investigators: Friedrich B. Prinz, Professor, Mechanical Engineering; Rainer Fasching, Research Associate, Eric Tao, Graduate Researcher, Seoung-Jai Bai, Graduate Researcher

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Objective: The hypothesis of this research assumes that advanced fabrication methods enable the creation of unique measurement devices, which may lead to new scientific insights regarding electrochemical reactions inside biological cells. The combination of Reactive Ion Etching (RIE) with focused ion beam milling and material deposition allows for the generation of a new class of electrochemical probes. Such probes are capable of mapping biochemical reactions inside cell membranes.

A pencil-shaped electrochemical transducer system for characterization of reduction oxidation potential in live biological cells with nanometer dimensions is being developed. High Aspect Ratio Silicon (HARS) tip structures are fabricated, combining isotropic and anisotropic deep RIE processes. In this way, aspect ratios of greater than 20, with tip radii of smaller than 50 nm can be achieved. The fabrication technology explored as part of this project will be able to perform electrochemical characterizations including cyclic voltammetry and impedance measurements.

Background: One key issue for the realization of the hydrogen economy is the economic production of hydrogen without producing CO_2 as a byproduct. In response to this challenge, Professors James Swartz and Alfred Spormann are pursuing the direct production of hydrogen via bio-conversion as part of GCEP's hydrogen initiative. To better understand the science of hydrogen production sensors are being developed in this project that will be capable of investigating reduction-oxidation potentials within the cytosol of genetically modified biological cells. Any sensor embedded into such cells needs to be at least one order of magnitude smaller than the diameter of the cell, which spans about three microns. These size constraints pose a significant challenge for today’s micro and nano fabrication technologies.

Approach: HARS tips are shaped combining isotropic etching with anisotropic Deep-RIE-silicon etching. This process is based on the ‘direct fabricated tip’ technique introduced for AFM probes (Boisen *et al.,* 1996). A 1 μm oxide layer is grown on a 500 μm thick wafer using wet oxidation. Next, oxide patterns (caps) for silicon tip-shaping are formed. Using an isotropic SF_6 based RIE, the top of the tip structure becomes shaped while retaining the silicon oxide cap. A Deep-RIE process is used to etch the shaft, exploiting the remaining silicon oxide caps as an etch mask. Using this process, an aspect ratio greater than 20 is achievable. Subsequently, wafers are thermally oxidized (Itoh *et al.,* 1995; Marcus *et al.,* 1990). Finally, the grown oxide is removed using a Buffered Oxide Etch (BOE) to release the oxide caps and form sharpened HARS tips. Repetitive oxidizing and BOE etching can lead to further sharpening of the tip (Marcus *et al.*, 1990; Folch *et al.,* 1997). Two sharpening cycles are routinely run to achieve tip radii smaller than 50 nm.

In a subsequent process, electrode tips are fabricated by patterning metal nano-electrodes on top of the silicon tips and subsequently connected to the bonding structures. The process begins with a Low Pressure Chemical Vapor Deposition (LPCVD) of a silicon layer with thickness of 200 nm. This layer serves as isolation between the metal layer and silicon substrate. Platinum is used as electrochemically active material. Platinum sputtering is being used to achieve good wall coverage and adhesion. Patterning of the metal layer is done with a lift-off technique. Next, a Plasma Enhanced Chemical Vapor Deposition (PECVD) silicon nitride layer is deposited onto the structured metal. Thick photo-resist is spun on in two steps in order to planarize the wafer surface. A RIE etching process is used to simultaneously etch back the resist and the silicon nitride layer on top of the platinum. The chemistry of the etch-back process is based on SF_6 and Freon 14 (CF_3Br). Platinum does not get etched by these etchants and is used as etch stop layer. In this way, exposure of the platinum on top of the tip in the sub micron range is achieved. Finally, the bonding pads are opened, exposing the metal contacts.

A detailed view of the top of the pencil probe is depicted in figure 1a. It shows the platinum tip with a 100 nm thick silicon nitride passivation layer. The tip radius is approximately 200 nm and significantly increased compared to the radius of the pure HARS tips as shown in figure1b.

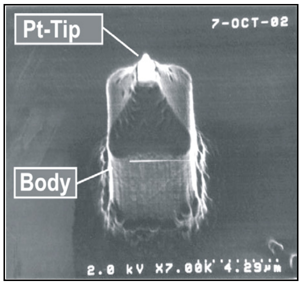

Figure 1a: Pencil Probe

Figure 1b: Pure HARS Tip

Multi-layer technology for electrical high-density connections between the two opposing sides of a wafer has been developed. This technology will be used to connect probes of an array to a signal processing CMOS circuit on the back side of the wafer. Openings in a double-sided polished wafer are created by applying a deep RIE technique. Hole structures with a diameter of 20 µm are formed through a 350 µm thick wafer. A multi-layer system of up to eight layers consisting of alternating conducting layers (N-type doped poly-silicon) and isolating layers (silicon-oxide) were grown until the vias were filled.

The applied LPCVD techniques guarantee a sufficient homogenous coating outside and inside of the entire structure to a minimum layer thickness of one µm. The connection quality has been examined combining impedance spectroscopy and Focused Ion Beam technology. Depending on the geometry and the doping profile of the poly-silicon layers, a connection resistance of less than 80 Ohms can be achieved with sufficient DC isolation. In this way, the multi-connection of up to four-isolated signal lines per opening was manufactured. This corresponds to a local connection density higher than 30,000/cm^2.

References

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Contact: Fritz Prinz, fbp@cdr.stanford.edu