IN SITU IMPEDANCE MONITORING OF IRIDIUM OXIDE ELECTRODEPOSITION FOR NEURAL RECORDING

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Abstract

Electrode-electrolyte impedance is one of important characteristic for neural recording due to its influence on the signal/noise ratio and signal distortion. Therefore lowering the electrode impedance brings benefits to recording of bio-signals. In this work, platinum electrodes are modified with Iridium Oxide (IrOx), a biocompatible metallic oxide film, aiming lowering the electrode impedance. It was used either potential control (potentiostatic) or current control (galvanostatic) processes to perform the electrodeposition as well as *in situ* impedance spectroscopy. The modified electrode impedance spectra was fitted by modifying the Randles model with a large capacitor, resembling the additional active area.

Keywords: electrode modification, impedance spectroscopy, neural recording, Iridium Oxide

Introduction

The electric contact between an electrode and living tissue has an electric impedance whose high values reduce signal to noise ratio and increase signal distortion. This is particularly relevant for microelectrodes due to their reduced dimensions and also because their impedance has the tendency to increase even more due to adsorption of biological material [1]

Simulations based on simple Randles model [2-4] shows that at microscale electrodes, the capacitive coupling is the major factor for the most physiological responses (mHz-kHz) and the electrode impedance increases as the electrode area decreases as well as its thermal noise (Johnson-Nyquist) (Figure 1).

Strategies to lower the impedance are of key importance in applications such as the recording of neural signals. Iridium Oxide (IrO_x), a biocompatible metallic oxide film, is an excellent candidate to promote this impedance reduction given its texture and because it is an electroactive material, i.e., it forms a fast-kinetics redox system on the electrode surface, therefore allowing Faradaic charge transfer 0.

In this work, platinum electrodes are modified with Iridium Oxide aiming lowering the electrode impedance. This was accomplished by directly electroplating over the platinum substrates. This method brings advantages such as depositing onto several geometries and morphologies (cavities), easy control of process parameters – mainly current and voltage - and hence material properties and finally it is a low cost process, especially if compared with sputtering. On the other hand, electroplating needs a conductive layer underneath although this can be overcome by using electroless or autocatalytic deposition processes.

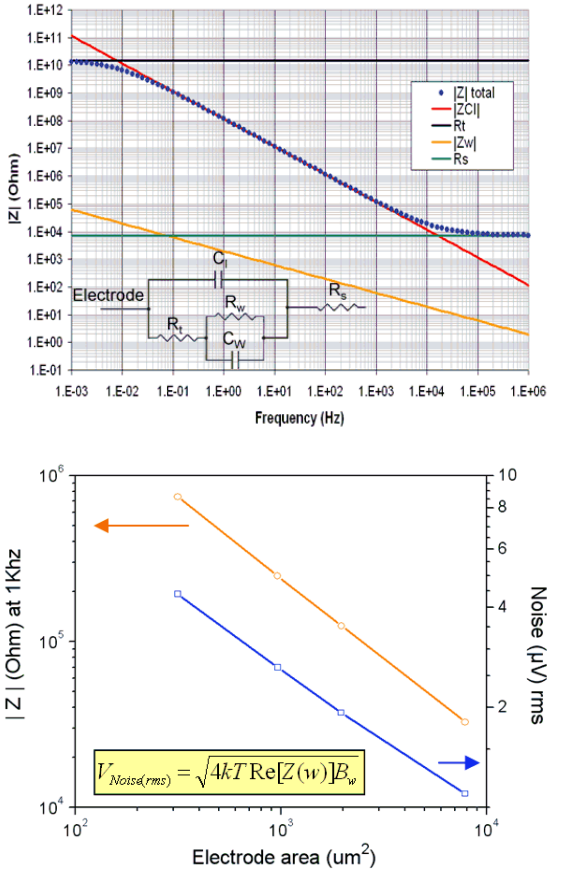

Figure 1: Platinum electrode impedance and thermal noise simulations based on Randles model.

Electrode Modification

IrO_x coatings have been obtained by directly electroplating over platinum electrodes. The electroplating solution is water based containing iridium chloride ($IrCl_4 \cdot H_2O$), hydrogen peroxide (H_2O_2), oxalic acid $(COOH)_2 2H_2O$ and potassium carbonate to adjust the pH to 10.5 0. Sample pretreatment is performed through surface degreasing with acetone followed by isopropanol and rinsing in deionized water. An additional treatment to improve adhesion can be done by slightly etching the platinum surface in *aqua regia* solution (3 HCl:1 HNO_3:2 H_2O) at 50℃ or by using the standard RCA organic silicon cleaning (1 NH_4OH:1 H_2O_2:5 H_2O) at 70℃.

It was used either potential control (potentiostatic) or current control (galvanostatic) processes to perform the electrodepositions, through three electrode setup (AgCl/Ag reference). We have utilized a CompactStat electrochemical analyzer (Ivium Technologies) to realize such depositions and to perform electrochemical impedance spectroscopy (EIS) characterization of the obtained electrodes.

We monitored the impedance evolution during the deposition, scanning at a very slow rate the voltage (1mV/s for potentiostatic deposition) or the current (10µA/s in galvanostatic deposition). Figure 2 shows a potentiostatic deposition over a 2 cm^2 $Pt/SiO_2/Si$ sample.

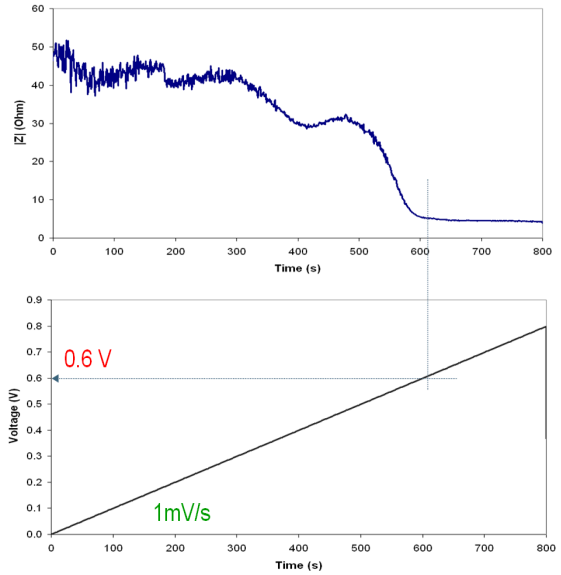

Figure 2. In situ potentiostatic deposition with impedance measurements over platinum sample A=2cm².

We observed that when the voltage reaches 0.6V causes a drop in the impedance evaluated at 10Hz. The same behavior can be obtained at current density of 2.5 A/m^2, figure 3. The impedance does not change within the layer thickness. This is because the surface roughness stays approximately the same for as long as the deposition conditions are not changed.

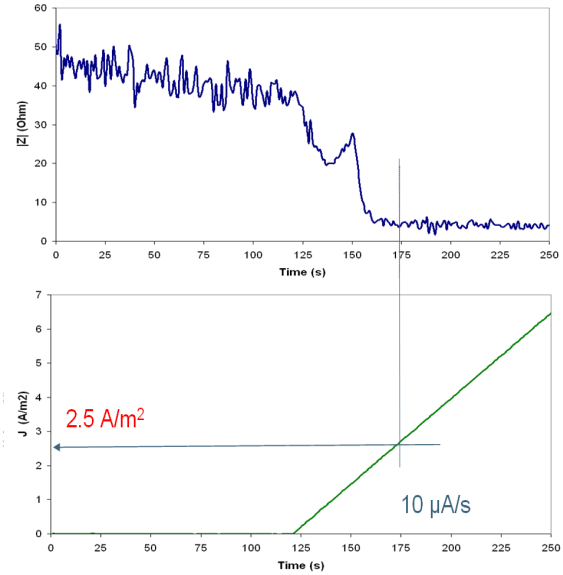

Figure 3. In situ galvanostatic deposition with impedance measurements over platinum sample A=2cm².

Using the known voltage and current range, it was performed several depositions schemes, including constant current and constant voltage, over platinum microelectrodes (Φ=100µm.). Constant current deposition leads to a similar decreases on the impedance, evaluated at 1 KHz – about 14 times - but has better performance over a large frequency range, figure 4.

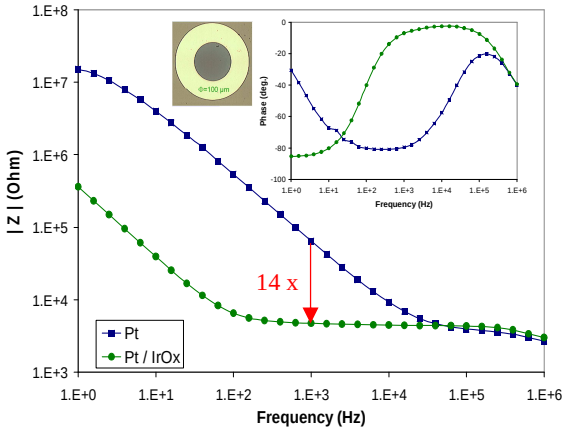

Figure 4: Impedance change after galvanostatic deposition of IrO_x over platinum electrode with 100 µm diameter.

A series of galvanostatic deposition at a constant current at different times lead to a conclusion that indeed the impedance drops right away since the first layer covers the platinum underneath, figure 5.

SEM analysis showed that depositions beyond a thickness of 200 nm leads to film cracks and further impedance decreasing due to exposition of the underneath platinum layer. Below that thickness very good uniformity was

achieved. AFM analysis indicated a surface roughness around 8 times greater than that for the base platinum electrode.

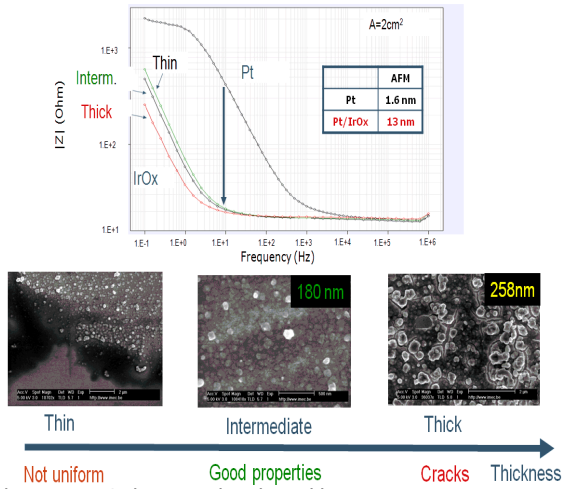

Figure 5. Galvanostatic deposition at constant current showing the film development over the thickness at platinum samples A=2cm².

Simulation of the modified electrode was achieved by modifying the previously reported Randles model for a platinum electrode [3], with a large capacitor in parallel, resembling the additional active area (200x) provided from the IrOx film, figure 6.

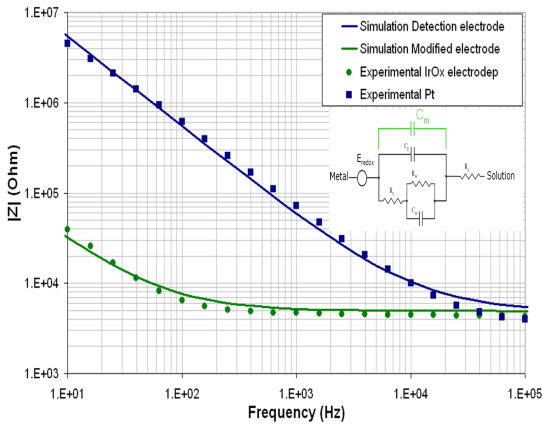

Figure 6. Impedance simulations and experimental data with and without IrOx covering a platinum microelectrode (Φ=50µm).

Conclusion

Modification of platinum microelectrodes using a biocompatible metallic oxide film – Iridium Oxide - successfully decreased their impedance by a factor of 14 at 1KHz. *In situ* Electrochemical Impedance Spectroscopy allowed determine the deposition window either for potentiostatic or galvanostatic processes. SEM images showed film cracks over 200nm thickness due to mechanical stress. A modified electrode-electrolyte model (Randles circuit), suggested an active area increasing over 200 times.

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