

## STUDY ON PULSED DC-SPUTTERING OF IRIIDIUM OXIDE FOR IMPLANTABLE ELECTRODES

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**Abstract** — A pulsed-DC reactive sputtering technique was used for iridium oxide thin-film deposition. Frequency, pulse width, oxygen flow and deposition time were changed over several deposition sessions, regarding implantable electrode applications. Surface and electrochemistry analysis were performed for deposition evaluation. The results evidenced that the deposited film sputtered with 2 sccm of oxygen flow and 50 KHz pulse frequency, achieved best surface structure and electrochemical properties. Impedance tests revealed an almost purely resistive behaviour for stimulation signal frequencies 0.1-10 KHz, with a value of 150  $\Omega$ .

**Key Words:** IrO<sub>x</sub> Sputtering, implantable electrodes.

### I INTRODUCTION

The study of brain organization and biophysical properties of the neurons have been demanding more sophisticated electrode fabrication technologies. Bio-potential electrodes, either used in functional medical implants or electroencephalogram (EEG) recordings [1] play an important role in this research. Iridium oxide (IrO<sub>x</sub>) coated electrodes have been very promising as stimulation electrodes due to their biocompatibility and suitable electrochemical properties. IrO<sub>x</sub> coatings have been deposited electrochemically from solution, reactive laser ablation and reactive sputtering [2]. IrO<sub>x</sub> coated electrodes are also being tested as recording electrodes for EEG [3].

Iridium oxide thin-films can be deposited through reactive sputtering whether the target is dc-powered [4] or rf-powered [2] in a plasma environment. Iridium oxide species were pulsed dc-sputtered as reported in this work. Pulsed power as opposed to dc-power prevents the formation of arcs [4]. DC-sputtering of dielectrics causes the inside walls of the deposition chamber to be coated with non-conducting layers that accumulate electric charges from the plasma. These charges create arcs that are responsible of particulates formation into the deposited films and power

supply damage. Therefore, a pulsed dc-signal is applied to a magnetron that is coupled on the target and traps the ionized plasma nearby. The power is applied to the target in a pulsed voltage sequence of “on-time” and “reverse-time” periods. During the “on-time”, a few hundred volts are applied to the target. A small positive voltage (around 20 V) is applied to the target during the “reverse-time”. The dielectric coated surfaces are charged up during “on-time” and discharged during the “reverse-time” [4]. Typically the “reverse-time” is 1/10 of the “on-time”.

The main purpose of this work is to evaluate the pulsed dc-sputtering technique performance for IrO<sub>x</sub> bio-potential electrodes. Surface structure analyses, charge delivery capacity (Q<sub>d</sub>) and impedance (Z) were analyzed regarding stimulation applications.

### II EXPERIMENTAL DETAILS

Several sputtering sessions with different parameter values were performed, however the film thickness was meant to be constant between sessions. Each session's parameter values, in Table I, were set based on previous DC-sputtering studies [5] and each pulsed session already done.

A Nordiko NS 2550 sputtering tool and a ENI RPG-100 pulse generator for plasma applications were used in IrO<sub>x</sub> thin film depositions. A MKS Baratron gauge was used to measure oxygen partial pressure and analytically infer about oxygen consumption by the growing film. All films were reactively sputtered in Ar/O<sub>2</sub> plasmas after a Ti adhesion layer deposition on the substrates. The film thickness was measured with a Tencor PA-10 profilometer. The sputtering chamber was evacuated to 10<sup>-6</sup> mbar by a cryogenic pump before every deposition. The system pumping speed was

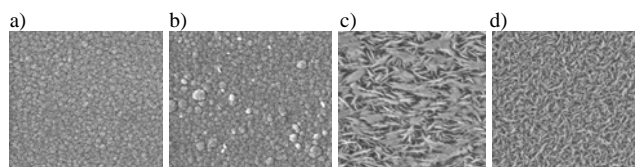
controlled with a throttle valve. In all sputtering sessions, the pulsed voltage sequence was applied to the target with a power of 180 W and the argon flow in the plasma was kept at 100 sccm.

**Table 1. Sputtering sessions' details**

Session	1	2	3	4	5	6	7
Frequency (KHz)	50	50	50	50	75	75	75
Pulse width (ns) "on-time"	1056	1056	1056	1056	3056	3056	3056
Oxygen flow (sccm)	2	3.5	6.5	10	2	3.5	6.5

### III RESULTS DISCUSSION

Figure 2 depicts SEM images of surface structures sputtered under 50 KHz pulse frequency for different oxygen flow values,  $Q(O_2)$ . On the one hand, under moderate oxygen flow conditions, images a) and b) of Fig. 2 show a grainier surface, on the other hand, upon further  $Q(O_2)$  increase, images c) and d) reveal increasing dendritic surfaces. This seems to be a transition phase between grainier and dendritic surface structures, which makes the  $IrO_x$  thin-film to loose coherence and become mechanically unstable.



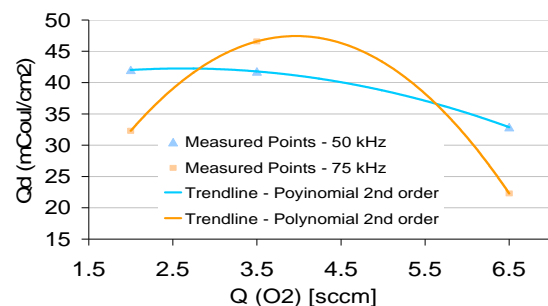
**Fig. 2. SEM images taken on samples sputtered surfaces at different oxygen supplies for 50 KHz pulse frequency. a) 2 sccm. b) 3.5 sccm c) 6.5 sccm d) 10 sccm.**

In fig. 3, the  $Q_d$  is plotted against oxygen supply to the chamber, after 50 cycles of activation, for both 50 (blue line) and 75 KHz (yellow line) pulse frequencies. The maximum  $Q_d$  occur around 2 sccm for the 50 KHz pulse frequency case. Electrochemical data from depositions with  $Q(O_2)$  values lower than 2 sccm need to be collected, as future work, in order to get stronger conclusions. However, having in account the support from previous studies [2], electrochemical activity can be said to rise with increasing  $Q(O_2)$  up to 2 sccm. This dependence can be due to the surface porosity shown in fig. 2 a) and b), since under moderate  $Q(O_2)$  the material matrix is more permeable to water and ionic species, leading to a higher charge delivery. The decline in  $Q_d$  can be related to the mechanical stability loss registered in pictures c) and d).

The variance of  $Q_d$  in respect to  $Q(O_2)$  for both 50 and 75 KHz is illustrated on fig. 3. The maximum

$Q_d$  for a sample sputtered with a pulse frequency of 75 KHz is achieved around 4 sccm of  $Q(O_2)$ . Therefore, increasing the pulse frequency may shift the maximum  $Q_d$  to higher  $Q(O_2)$  values. The  $Q(O_2)$  value correspondent to the maximum  $Q_d$ , for 50 KHz pulsed sputtering, roughly matches the  $Q(O_2)$  of the best surface structure detected in fig. 2. Hence the best surface structure for a higher frequency sputtering deposition is expected to occur at a higher  $Q(O_2)$  value.

Impedance value decreases with activation. After 50 cycles of activation, the impedance value for both cases a) and b) in fig. 2 is around 150  $\Omega$  and nearly resistive for the frequency range 0.1-10 KHz.



**Fig. 3.  $Q_d$  after 50 cycles of activation versus oxygen flow for 50 and 75 KHz pulse frequencies.**

### REFERENCES

- [1] Patrick Griss, Peter Enoksson, Heli K.Tolvanen-Laakso, Pekka Merilainen, Stig Ollmar, and Goran Stemme, Micromachined Electrodes for Biopotential Measurements, *J. Microelectromechanical System*, **10**, 1, pp.10-16, 2001.
- [2] B.Wessling, W.Mokwa, and U.Schnakenberg, RF-Sputtering of iridium oxide to be used as stimulation material in functional medical implants, *J. of Micromechanics and Microengineering*, **16**, pp.142-148.
- [3] J.H.Correia, B.Wessling, N.S.Dias, H.R.Silva, W.Mokwa, and U.Schnakenberg, Sputtered TiN and IrO<sub>2</sub> Electrodes Versus Standard Ag/AgCl Electrodes for Non-Invasive EEG Applications, *MME Digest*, **1**, pp.25-28, 2006.
- [4] A.Belkind, A.Freilich, J.Lopez, Z.Zhao, W.Zhu, and K.Becker, Characterization of pulsed dc magnetron sputtering plasmas, *New J. of Physics*, **7**, pp.1-16, 2005.
- [5] E.Slavcheva, R.Vitushinsky, W.Mokwa, and U.Schnakenberg, Sputtered iridium oxide films as charge injection material for functional electrostimulation, *J. Electrochem. Soc.*, **151**, E2, pp.26-37, 2004.