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Intrinsic Activation of Iridium Electrodes over a Wireless Link

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***Abstract*— Activated Iridium Oxide Film (AIROF) microelectrodes are regarded as advantage for stimulation of neural tissue owing to their superior charge injection capabilities, as compared to other noble-metal based electrodes. Including AIROF electrodes within an implantable neural stimulator can be challenging since the stimulator fabrication steps often involve elevated temperatures at which the AIROF can be damaged. In this work, a wireless neural stimulator application-specific-integrated-circuit (ASIC) was used to intrinsically activate iridium microelectrodes. This intrinsic activation allows for the growth of the AIROF as the final assembly step after the entire device is assembled, thus avoiding stress on the AIROF. Since a typical neural stimulator is essentially a current-controlled driver with voltage compliance limits, its output waveform can be tuned to match the traditional voltage pulsing/ramp activation waveform. Here the feasibility of the current driven activation of iridium electrodes, over a wireless link, is demonstrated.**

I. INTRODUCTION

The intracortical visual prosthesis project (ICVP) at the Illinois Institute of Technology includes a wireless implantable neural stimulator module that is specifically designed for delivery of charge through intracortical electrodes, towards the goal of restoring visual sensation in those individuals with blindness [1]. The wireless stimulator module consists of an ASIC chip fabricated in the XFAB CX08 BICMOS process, a miniature coil for transcutaneous powering and communication, and sixteen AIROF microelectrodes with surface area of $2000\,\mu m^2$. AIROF microelectrodes have been widely studied and used for neural stimulation applications [2][3]. They are capable, relative to bare metal electrodes, of higher charge capacity due to the reversible Ir(+III)/Ir(+IV) redox reaction mechanism. To protect the oxide film and the biological media from irreversible electrochemical effect, such as water electrolysis, during the charge delivery, the water window compliance limit technique has been widely adopted by many implantable neural stimulators that drive the microelectrodes [4].

The ICVP ASIC chip is equipped with 16 compliance-limited independent current driver that can be commanded wirelessly to generate the phosphene-based neural stimulation pattern for the intracortical visual prosthesis. Interestingly, this same stimulator architecture can also provide an extra functionality, that is, to activate iridium microelectrodes *in situ*.

The ICVP stimulator module is protected from the deleterious effects of body fluids by a silicone polymer encapsulation. As part of the final encapsulation process, the module must be subjected to temperatures of up to 150 °C for over 1 hour. Within our laboratory it has been confirmed that because the AIROF is a hydrated structure, at temperatures above 60 °C the AIROF can be irreversibly damaged due to mechanical stresses within the film during water evaporation. Within the stimulator module, the iridium metal microelectrodes are connected to the ASIC chip without any alternative electrical connections. Therefore the electrodes need to remain inactivated until the final elevated-temperature cure cycle is completed. Similar problems can be expected to occur in many stimulator designs that use AIROF electrodes since rapid loss of hydration places the AIROF at risk, and elevated temperatures can result from polymer curing or vacuum bake-outs.

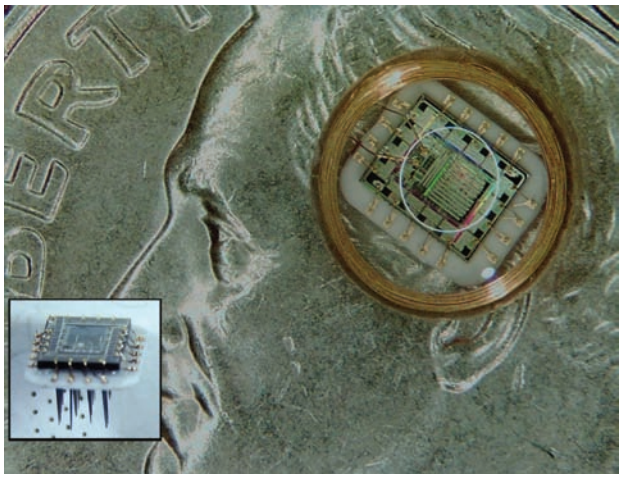

Fig. 1. Wireless Visual Prosthesis Stimulator Module

One obvious option for eliminating the temperature-induced stress upon the AIROF is to activate the bare iridium electrodes as the final assembly step, after all elevated temperature assembly steps have taken place. For a wireless stimulator, this requires that the 16 microelectrodes need to be activated by the ASIC chip itself. Unlike other noble metals such as Ru or Ta, holding the iridium metal at a constant potential, with respect to (WRT) the surrounding electrolyte won't cause its oxide film to grow. To form iridium oxide on an iridium metal electrode, the electrode voltage is typically ramped or pulsed between upper and lower limits . Though there is little difference in the chemical composition of the oxide film formed, the morphology of the film can be very different, depending upon the acid vs. base growth media [5], as well as the shape of the voltage waveform during the activation process [6]. According to

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the model suggested by [6], a compact oxide layer is first formed whose surface is then hydrated. The hydrated outer layer prevents the inner compact oxide from further hydration when the electrode voltage is held anodic. But it is also more difficult to reduce the outer layer, rather than the compact layer, when the electrode voltage is held cathodic. To accumulate the hydrated layers, the inner compact layer needs to be reduced and re-oxidized, such that a new hydrated "outer" layer is formed, thus adding to the volume of the total hydrated layer. Since the film growth depends on the dynamics of these intricate reactions in a seemingly paradox direction, the control of the activation voltage waveform and its timing become very important. It is also known that too many activation cycles could cause the AIROF film to delaminate (the aging effect) [7] and become mechanically unstable, most likely because the film is growing from inside out, and mechanical stress increases as the outer layers are subjected to the volume increases of the underlying growth.

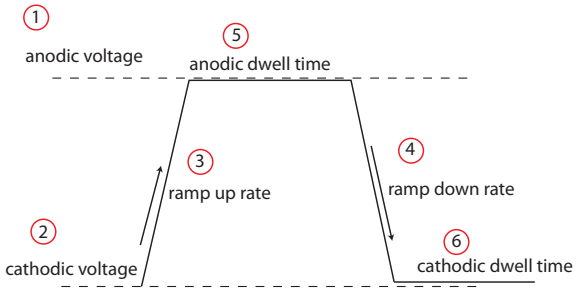

Fig. 2. Electrode Voltage Waveform for AIROF Activation

The ICVP stimulator module was originally designed as a neural stimulation constant current generator with precise control of the stimulation current, and its pulse width via commands sent over a wireless inductive link operating at 5 MHz and 1.2MB/second. Within command set of the module, additional commands are provided to suspend the pulse-width timer clock, and to update the stimulation current output in real-time, thus allowing the ASIC to operate as an arbitrary current waveform generator. Using anodic and cathodic compliance limits that are appropriate for the growth of AIROF, the ASIC is able to mimic the typical AIROF voltage activation waveform as shown in Fig. 2.

Here we report on the use of the ICVP ASIC for the first-reported demonstration of wireless activation of iridium electrodes for a neural prosthesis.

II. MATERIAL AND METHODS

A. Bare Iridium Metal Electrodes

Pure iridium metal electrodes either in single unit form or 16-electrode array form were purchased from Microprobe for Life Sciences Inc. (Gathersburg, MD). Each individual electrode was etched from the solid iridium wire using a controlled etching protocol in order to optimally shape the electrode for insertion into the neural tissue. The entire electrode was coated with Parylene-C (poly-dichloro-diparaxylene). Then a dual-beam excimer laser was used to expose the surface area at the tip of the electrode. In our case, the exposed geometric area of the electrode is approximately 2000 μm^2 with about a 10% variation between electrodes. A microscopic imaging method was used to calibrate the shape of the electrode and verify its surface area in our lab. The received electrodes were subject to ultrasonic and oxygen plasma chamber cleaning to remove residual surface debris resulting from the laser ablation.

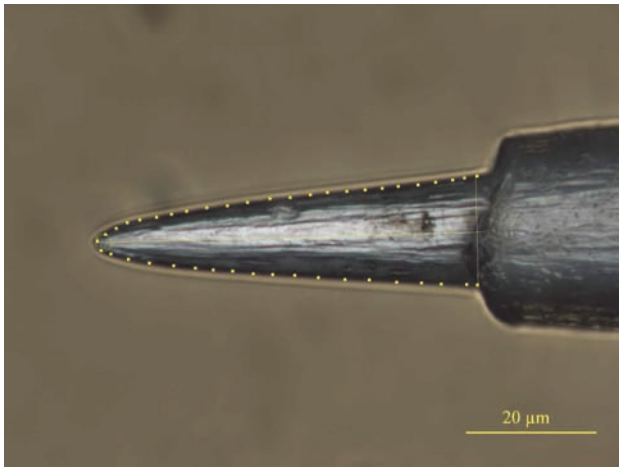

Fig. 3. Bare Iridium Metal Microelectrode Microscope Photo

B. Electrode Characterization

A three-electrode electrochemical cell was used with a Ag/AgCl reference electrode and a large Pt mesh as the counter electrode in PBS solution at room temperature (~25 °C). Cyclic voltammetry (CV) was measured at a 50 mV/s sweep rate within phosphate buffered saline (PBS) using a Gamry Potentiostat. The current pulsing and voltage measurements were made with the neural stimulator and a 100 Ω resistor in series with the counter electrode in the current return path. A Tektronix oscilloscope was used to record the waveforms.

C. Electrode Activation

We tuned the activation parameters previously optimized for our 2000 μm^2 microelectrodes as follows:

- Anodic voltage: 0.9 V to 1.0 V vs. Ag/AgCl
- Cathodic voltage: −0.6 V to −0.8 V vs. Ag/AgCl
- Ramp up/down rate: 15 to 30 V/s
- Anodic/cathodic dwell time: 15 seconds

In the final electrode array assembly, there is no Ag/AgCl reference electrode. Two longer stabilizing Pt-Ir electrodes serve as the reference and counter electrodes respectively. As shown in the setup diagram (Fig. 4), a large Pt mesh counter electrode in the cell was connected to the counter

and the reference terminals of the ASIC, simulating the Pt-Ir stabilizing electrodes. The working iridium metal electrode was connected to one of the 16 channels of the ASIC.

Since the open circuit potential of Pt is approximately 0.2V above Ag/AgCl, the electrode activation voltage is shifted downward accordingly.

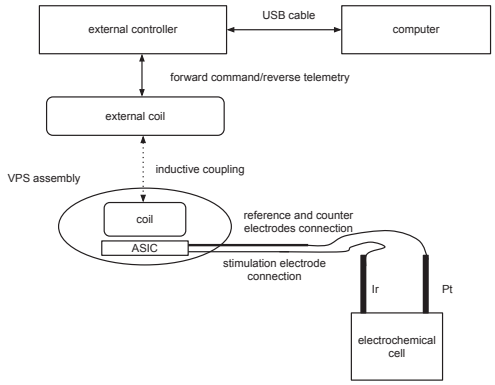

Fig. 4. Wireless ICVP Current Driven Activation System Diagram

After activation, a Ag/AgCl electrode was inserted into the cell and the single electrode was disconnected from the wireless ASIC system, and connected to the Gamry for CV characterization.

III. RESULTS AND DISCUSSIONS

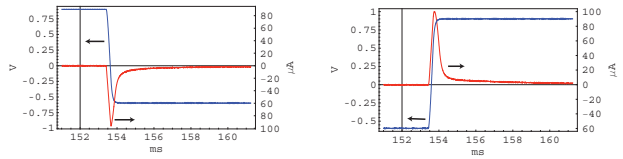

Fig. 5. Voltage and Current Waveform of the Gamry Voltage Driven Activation

As shown in the current and voltage plot (Fig. 5), when the square voltage pulse (from the Gamry system) is used to activate the electrode, since only the voltage levels and pulse duration are specified, the electrode current is allowed to peak to very large value, i.e. 100 μA in this case for both anodic and cathodic phases.

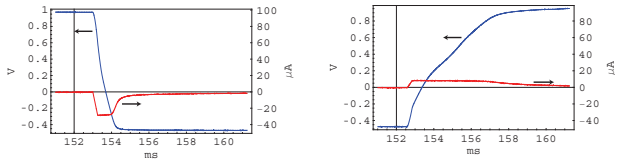

Fig. 6. Voltage and Current Waveform of Stimulator Current Driven Activation

On the other hand, when using the wireless ASIC for activation, the electrode drivers are restricted to constant-current operation. Based upon empirical measurements in our laboratory, we set the cathodic current to be -20 μA and the anodic current at 10 μA. The resulting voltage ramps for the anodic and cathodic phases are shown in Fig. 6.

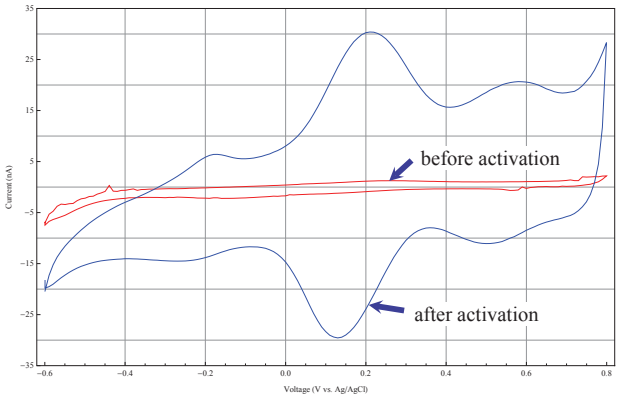

Fig. 7. CV Waveform of an Iridium Electrode Before and After Activation

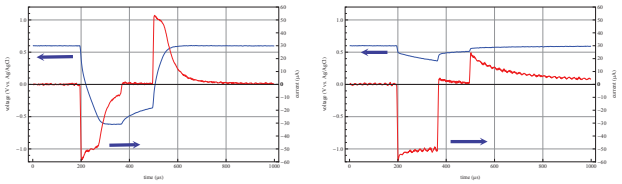

Fig. 8. Voltage and Current Pulsing Waveform of an Iridium Electrode Before and After Activation

Using the current driven protocol, Fig. 7 shows the CV waveform of an iridium electrode prior to, and following the activation. We were able to make the measurement because we allowed for physical separation of the electrode from the wireless ASIC in this experimental setup. The feature peaks in the CV loop and the CSC_c (time-integral of the cathodic current in one voltage cycle) value clearly indicates the growth of AIROF film that are similar to the results of a voltage-driven activation.

Fig. 8 shows the current pulsing waveform before and after activation. It is obvious that before activation (Fig. 8 left), the bare iridium metal electrode can not sustain 50 μA cathodic current before the cathodic voltage excursion saturates at the water window (-0.6 V), which causes the ASIC to cut back the current. After activation (Fig. 8 right), the same electrode can deliver 50 μA with only a small cathodic voltage polarization. This inter-phase voltage, i.e., the electrode voltage during the cathodic-to-anodic current phase transition, when the current is zero, clearly shows the distinction between an inactivated and activated iridium electrode.

Our typical protocol is to measure CV on bare iridium electrode prior to activation so that we have a baseline measurement to be compared with the CV after activation.

In the real stimulator module assembly, there is certainly no possibility to perform CV measurements. However, using the wireless link and the reverse-telemetry capability to transmit the electrode voltage waveform during a stimulation pulse, we can easily determine whether the electrode is activated or not by observing the electrode voltage waveform during the normal constant-current pulsing.

We observed that if we do not perform CV on the bare iridium metal prior to activation, it is very difficult to start the oxide film growth using the fore-mentioned current driven protocol. However if we mimic the behavior of a CV measurement, using the wireless ASIC, by dwelling the electrode voltage between ± 0.6 V for several cycles, the oxide film can start to grow. Interestingly, this initial voltage cycling step is not required for voltage driven activation, with or without a pre-activation CV.

We hypothesize that as suggested by [6], the inner compact oxide layer needs to be "shielded" by the outer hydrated layer. Otherwise it can be quickly oxidized, but also quickly reduced, resulting in no net film growth. Our ± 0.6 V initial dwelling procedure may serve as a way to retain and hydrate this inner compact layer to form the first hydrated iridium oxide layer, which we refer to as the "seed" layer. It is unknown why the voltage driven activation method, which essentially masks different steps of the reaction kinetics, can surpass the "seed" layer unaffected.

Noted that the CSC_c value of AIROF microelectrodes is not the main objective for activation, since most of the charge capacity may not be fully utilized in the *in vivo* environment. The key is to engineer a film that has good mechanical stability and fast charge delivery kinetics.

IV. CONCLUSION

The activation of iridium electrodes using a wireless constant-current stimulation ASIC has been demonstrated. The significance of this accomplishment is that during the fabrication of implantable devices, electrodes are subjected to elevated temperatures that prove destructive to AIROF. The capability to activate as the last fabrication steps avoids the deterioration of the AIROF. Wireless growth of AIROF may provide an important capability for fabrication of miniature stimulators, including wireless silicon probes, for use in emerging neural prostheses.

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