In Vitro Comparison of the Charge-Injection Limits of Activated Iridium Oxide (AIROF) and Platinum-Iridium Microelectrodes

Stuart F. Cogan*, Philip R. Troyk, Julia Ehrlich, and Timothy D. Plante

***Abstract*—The charge-injection limits of activated iridium oxide electrodes (AIROF) and PtIr microelectrodes with similar geometric area and shape have been compared *in vitro* using a stimulation waveform that delivers cathodal current pulses with current-limited control of the electrode bias potential in the interpulse period. Charge-injection limits were compared over a bias range of 0.1–0.7 V (versus Ag|AgCl) and pulse frequencies of 20, 50, and 100 Hz. The AIROF was capable of injecting between 4 and 10 times the charge of the PtIr electrode, with a maximum value of 3.9 mC/cm^2 obtained at a 0.7 V bias and 20 Hz frequency.**

***Index Terms*—Charge-injection, current pulsing, iridium oxide, microelectrode, neural stimulation, platinum-iridium.**

I. Introduction

Microelectrodes for neural stimulation are typically characterized by a charge-injection limit that represents the maximum charge that can be injected into tissue without exceeding some predefined limits, within which the electrode is considered to operate reversibly. Reversibility in this context is taken to mean that the reduction and oxidation reactions mediating the charge transfer across the electrode-tissue interface can be reversed without the accumulation of electrolysis byproducts from the electrode or the physiologic environment. This reversibility is achieved with the use of charge-balanced waveforms which can take a variety of forms that include voltage as well as current control [1]–[3]. The use of voltage-controlled pulses without active charge balance has also been suggested as appropriate for stimulation in some circumstances [4]. The anticipated charge-injection requirements for stimulation of the central nervous system exceed the established limits of noble metal electrodes [5], and most studies involving intracortical stimulation employ activated iridium oxide electrodes (AIROF) that provide significantly higher levels of reversible charge-injection [6]–[8].

To access the full charge-injection capabilities of AIROF it is necessary to bias the electrode to a potential more positive than its equilibrium potential. With this strategy, the charge-injection capacity of AIROF can be increased from about 1 mC/cm^2 without bias to 3.5 mC/cm^2 at an 0.8 V bias using an 0.2 ms pulse width [6]. A positive potential bias may also be used to increase the charge-injection limits of Pt electrodes and charge-injection levels for cathodal pulsing of 0.6 mC/cm^2 with 0.2 ms pulses have been reported for an 0.8 V bias versus a saturated calomel reference electrode [9]. In the present work, the charge-injection limits of a AIROF and a PtIr-alloy microelectrode have been evaluated as a function of bias potential in response to cathodal-first current pulsing. The current waveform consisted of a leading monophasic cathodal pulse with a charge balancing anodal phase that reestablishes the desired bias level without exceeding the positive potential limit for oxidation of water on the AIROF or PtIr electrode. The microelectrodes had similar shape and geometric area allowing a direct comparison of the charge-injection limits as a function of bias.

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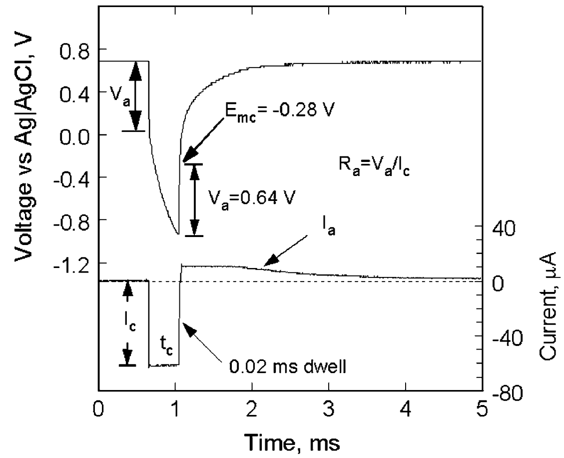

Fig. 1. Cathodal-first current pulse and corresponding voltage transient for an AIROF microelectrode subjected to a $I_c = 50\ \mu A$, $t_c = 0.4$ ms pulse at a bias of 0.7 V. The $Q_{inj} = 2.3$ mC/cm^2 polarized the AIROF to a $E_{mc} = -0.28$ V. The anodic current (I_a) and current-limit used to establish the positive bias after the cathodal phase are also shown.

II. Experimental

The iridium and PtIr (20% iridium) microelectrodes were fabricated at the Huntington Medical Research Institutes from iridium or PtIr wire shafts, $\sim$5 mm long and 35 μm in diameter. One end of each shaft was etched electrolytically to a cone terminating in a blunt, hemispheric tip approximately 12 μm in diameter with an included angle of 9°–11°. Electrical connection to the iridium was made by soldering the unetched end of the shaft into a 5-cm length of stainless steel tubing. The shaft and tubing were insulated with Parylene-C and a charge-injection site created at the distal tip by laser ablation. The exposed geometric surface area (GSA) of the electrodes was calculated from tip dimensions measured from photographs taken with an optical microscope. For this study, the GSAs of the iridium and PtIr electrodes were $877 \pm 100\ \mu m^2$ and $1105 \pm 100\ \mu m^2$, respectively. The iridium electrode was activated to AIROF by potentiodynamic pulsing between -0.6 and $+0.85$ V versus Ag|AgCl in phosphate-buffered saline (PBS) having a concentration of 0.13 M NaCl, 0.022 M $NaH_2PO_4 \cdot H_2O$, and 0.081 M $Na_2HPO_4 \cdot 7H_2O$ (pH $\sim$ 7.3). Cyclic voltammetry (CV) and potential transient responses during pulsing were measured in a three-electrode cell comprising a Ag|AgCl reference electrode, a large-area Pt mesh counterelectrode, and the AIROF or PtIr microelectrode. All potentials are with respect to Ag|AgCl unless noted otherwise. CVs were acquired with a computer-controlled Gamry PC4 potentiostat using manufacturer-supplied software. All CVs were measured at a 50 mV/s sweep rate between potential limits of -0.6 V and 0.8 V, beginning at open-circuit potential and sweeping in the positive direction first. Current pulsing was performed with a custom-built stimulator that provides cathodal current pulses with the electrode biased to a set potential, versus the reference electrode, in the interpulse period. Following the cathodal pulse, the bias is reestablished using an anodic recharge current source that maintains the electrode at the desired bias voltage. The anodic recharge current source inherently limits the electrode anodic potential by virtue of its own compliance supply

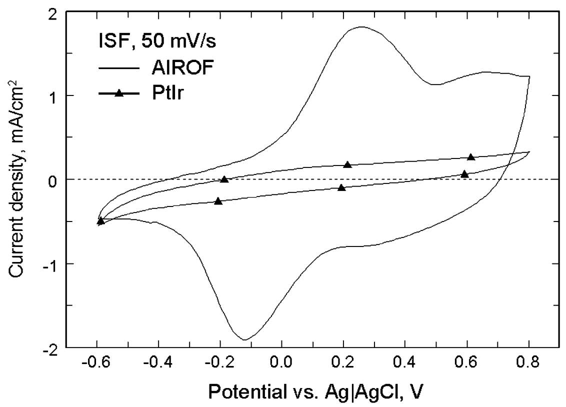

Fig. 2. Cyclic voltammograms of AIROF and PtIr microelectrodes in ISF at a sweep rate of 50 mV/s.

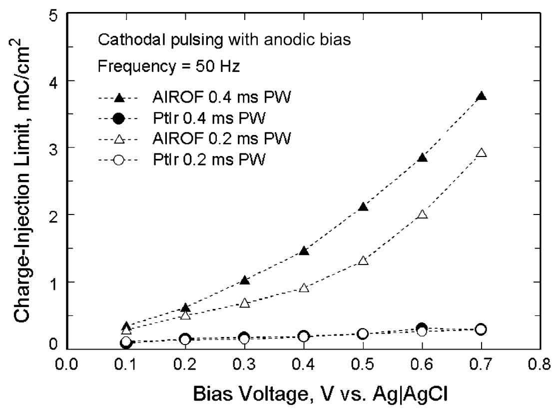

Fig. 3. Comparison of the charge-injection limits of AIROF and PtIr as a function of bias and pulse width.

that is set to the desired electrode bias. This voltage is derived using operational amplifier circuitry and is controlled relative to the Ag|AgCl reference electrode. Cathodal pulse widths of 0.2 ms and 0.4 ms, and pulse frequencies of 20, 50, and 100 Hz were investigated.

The potential transients were recorded with an oscilloscope and the maximum negative potential excursion (E_{mc}) calculated by subtracting the access voltage (V_a), associated with the ohmic resistance of the electrolyte and concentration polarization, from the maximum negative voltage in the transient. Usually, E_{mc} is equal to the electrode potential immediately after the end of the cathodic current pulse when V_a is zero. An example of a current and corresponding voltage waveform identifying E_{mc} is provided in Fig. 1. The maximum charge-injection limit of the AIROF and PtIr was defined as that which polarized the film to the potential for water reduction ($E_{mc} = -0.6$ V). The current pulsing was performed in a model interstitial fluid (ISF) electrolyte with a composition: NaCl 110 mM, $NaHCO_3$ 28 mM, $KHCO_3$ 7.5 mM, Na_2HPO_4 -$7H_2O$ 2 mM, and 0.5 mM each of NaH_2PO_4 -H_2O, $MgSO_4$, $MgCl_2$, and $CaCl_2$. A pH of 7.4 was maintained by a gentle flow of 5% CO_2/6% O_2/89% N_2 gas through the electrolyte. The measurements were made at room temperature.

III. Results and Discussion

Cyclic voltammograms of the AIROF and PtIr microelectrodes are compared in Fig. 2. The cathodic charge storage capacities (CSC_c) of the electrodes, calculated from the time integral of the cathodic current during a 50 mV/s CV were 23 mC/cm^2 and 5.6 mC/cm^2 for the AIROF and PtIr, respectively. The CV of the AIROF in ISF has a shape that is different from those observed in electrolytes with high, nonphysiologic buffer concentration, which produce more pronounced Ir^{3+}/Ir^{4+} reduction-oxidation peaks. The measured CSC_c of the PtIr electrode is also higher than typically observed in deaerated electrolytes due to a contribution from reduction of dissolved oxygen in the ISF.

The charge-injection limits for pulsing the AIROF and PtIr microelectrodes with 0.2 ms and 0.4 ms cathodal pulse widths at a frequency of 50 Hz are shown in Fig. 3. The maximum Q_{inj} for the AIROF at 50 Hz was 3.8 mC/cm^2 at a bias of 0.7 V and 0.4 ms pulse-width. The Q_{inj} at the 0.4 ms pulse-width decreased monotonically to 0.35 mC/cm^2 as the bias was decreased to 0.1 V. The 0.1 V bias level is typical of the open-circuit potential of AIROF observed *in vivo* in cortex, and emphasizes the necessity of using a positive bias with AIROF electrodes if they are to be employed at high levels of charge injection. The Q_{inj} of the PtIr showed the same bias dependence, decreasing from 0.3 mC/cm^2 at 0.7 V to 0.09 mC/cm^2 at 0.1 V for a 0.4 ms pulse-width. The AIROF Q_{inj} decreased with increasing pulse frequency at the 20, 50, and 100 Hz levels investigated, decreasing by 38% for a 0.2 ms pulse-width as the frequency was increased from 20 Hz to 100 Hz. The charge-injection limits for the AIROF and PtIr are compiled in Table I as a function of frequency and pulse-width for a fixed bias of 0.7 V. From a comparison of these data, it is apparent that a $CSC_c = 23$ mC/cm^2 AIROF microelectrode has a factor of $\sim$10 greater Q_{inj} than PtIr at a 0.7 V bias. This advantage decreases at less positive bias levels and, at a 0.1 V bias, the AIROF Q_{inj} was only 3 and 4 times greater than PtIr for 0.2 ms and 0.4 ms pulse widths, respectively. The charge-injection data in Fig. 3 are representative single trials and some variation in Q_{inj} is expected between nominally identical electrodes because of inexact areas and subtle differences in the shape of the charge-injection tips. Employing an anodic bias with a multi-channel array of microelectrodes will also introduce complexity in the stimulation circuitry. Using integrated circuit design techniques, several methods by which compliance-supply limited current sources can be used to maintain the electrode bias during the interpulse interval when stimulating with multi-channel systems have been identified. However, the successful implementation of such systems in hardware remains to be demonstrated.

TABLE I
Charge-Injection Limits of AIROF and PtIr Microelectrodes as a Function of Pulse Width and Frequency at an 0.7 V Bias

	Charge-injection limits, mC/cm^2			
	AIROF pulse-width, ms		PtIr pulse-width, ms	
Frequency, Hz	0.2	0.4	0.2	0.4
20	3.4	3.9	0.29	0.3
50	2.9	3.8	0.30	0.29
100	2.1	3.2	0.27	0.22

IV. Conclusion

The charge-injection limits of AIROF and PtIr microelectrodes with similar geometric area and shape have been compared in a model that closely matches the inorganic electrolyte content of ISF. A stimulation waveform that delivers cathodal-current pulses with current-limited electrode potential control in the interpulse period was used in the study. The AIROF was capable of injecting between 4 and 10 times the charge of the PtIr electrode, using a criterion that the maximum negative potential excursion of the electrode should not be driven more negative than -0.6 V (Ag|AgCl), the limit for avoiding reduction of water at the electrode. The Q_{inj} of AIROF increased with increasing bias and decreasing pulse frequency, with a maximum value of 3.9 mC/cm^2 obtained at 0.7 V and 20 Hz. To obtain the maximum charge-injection levels provided by AIROF electrodes, it is necessary to employ a stimulation protocol and hardware that permits the use of a positive bias during pulsing. Consideration must also be given to the frequency at which the pulses are delivered, since a notable reduction in the charge-injection limit was observed as the frequency was increased from 20 Hz to 100 Hz.

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Recording Human Evoked Potentials That Follow the Pitch Contour of a Natural Vowel

Hilmi R. Dajani*, David Purcell, Willy Wong, Hans Kunov, and Terence W. Picton

***Abstract*—We investigated whether pitch-synchronous neural activity could be recorded in humans, with a natural vowel and a vowel in which the fundamental frequency was suppressed. Small variations of speech periodicity were detected in the evoked responses using a fine structure spectrograph (FSS). A significant response ($P \ll 0.001$) was measured in all seven normal subjects even when the fundamental frequency was suppressed, and it very accurately tracked the acoustic pitch contour (normalized mean absolute error $< 0.57\%$). Small variations in speech periodicity, which humans can detect, are therefore available to the perceptual system as pitch-synchronous neural firing. These findings suggest that the measurement of pitch-evoked responses may be a viable tool for objective speech audiometry.**

***Index Terms*—Bioelectric potentials, biomedical signal processing, speech processing, time-frequency analysis.**

I. INTRODUCTION

Objective audiometric tests that use clicks, tone bursts, or steady state tones give only limited information about a subject's ability to perceive speech [1]–[3]. Consequently, there is an interest in developing objective hearing tests in which speech is the stimulus signal. In speech, pitch is the primary determinant of prosody, which includes linguistic, emotional, and speaker-specific information [4]–[6]. The speech signal has a fundamental frequency F0, the inverse of its basic periodicity, whose evolution over time is commonly referred to as the F0 or pitch contour. Previous studies have reported the recording of potentials evoked by natural and synthetic speech [7]–[9], and have described a peak in the spectrum of the response at F0. Krishnan *et al.* [10] have recently detected evoked responses that closely followed gross directional changes of around 10–50 Hz in the pitch contours of synthetic lexical tones of Mandarin Chinese, using a periodicity detection short-term autocorrelation algorithm [11].

The ear is very sensitive to small changes in F0 [4], the difference limen for (synthetic) vowels being in the range of 0.3% to 0.5% F0 for the male voice [12]. In this paper, we tracked evoked responses that closely followed fine variations of the order of a few Hertz in the F0 of a natural vowel using a fine structure spectrograph (FSS) [13]. This time-frequency analyzer allows the detection of small envelope and frequency variations in modulated signals. As the physiological mechanisms underlying pitch perception remain uncertain [14], the measurement of pitch-evoked responses in the electroencephalogram

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