

Effective Area and Charge Density of Iridium Oxide Neural Electrodes



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ABSTRACT

The effective electrode area and charge density of iridium metal and anodically activated iridium has been measured by optical and electrochemical techniques. The degree of electrode activation could be assessed by changes in electrode colour. The reduction charge, activation charge, number of activation pulses and charge density were all strongly correlated. Activated iridium showed slow electron transfer kinetics for reduction of a dissolved redox species. At fast voltammetric scan rates the linear diffusion electroactive area was unaffected by iridium activation. At slow voltammetric scan rates, the steady state diffusion electroactive area was reduced by iridium activation. The steady state current was consistent with a ring electrode geometry, with lateral resistance reducing the electrode area. Slow electron transfer on activated iridium would require a larger overpotential to reduce or oxidise dissolved species in tissue, limiting the electrodes charge capacity but also reducing the likelihood of generating toxic species *in vivo*.

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1. Introduction

Electrodes are used to stimulate excitable cells for the treatment of disease and to provide sensory cues. Charge must be delivered from the electrode to the target cells to induce an electrophysiological response. Stimulation of neurons can be performed in the peripheral or central nervous system. Flexible thin film electrocorticography (ECoG) electrode arrays are being implanted on the surface of the brain [1], microelectrode arrays are being implanted into the cortex [2] and macroelectrodes are being used for deep brain stimulation (DBS) [3]. Different electrode geometries are required to target various brain structures; for instance, ECoG and cortical electrodes may target the motor cortex to stimulate muscle movement in the body or the auditory cortex to elicit sound perception; while DBS electrodes are usually placed in the subthalamic nucleus.

To deliver charge from the electrode to the target neurons, an oxidising or reducing current must be applied at the electrode-tissue interface. For safe use, the applied current must not generate potentials that can breakdown water or corrode the electrode [4]. Platinum is used in clinical electrodes due to its high biocompatibility, biostability and relatively wide potential window. The charge injected from platinum is mostly from capacitance. Charge

generated by capacitance is proportional to the electrode area. Delivering sufficient charge from an electrode to stimulate a neuron is therefore dependant on electrode area. DBS electrodes stimulate a large region of tissue, and so the electrode area can be large. However cortical electrodes that try to target individual or limited groups of neurons must be much smaller. Microelectrodes that only deliver capacitive charge are unable to stimulate neurons without applying excessive potentials, leading to tissue and electrode damage.

To increase the charge capacity of electrodes, the use of Faradaic charge injection mechanisms is being utilised. A wide variety of doped conducting polymers have been applied to electrodes to increase the charge injection capacity of microelectrodes [5–7]. Changes in surface roughness and contact angle have also been found to affect neurite growth and proliferation [8,9]. This may allow directed growth of neurons to the electrode for improved chronic performance. Unfortunately the stability of doped conducting polymers is poor, delaminating from the underlying electrode or degrading [10,11].

Activated iridium is a conductive iridium oxide that undergoes a series of redox reactions of the general form $\text{IrO}_x(\text{OH})_y + z\text{H}^+ + ze^- \rightleftharpoons \text{IrO}_{x-y}(\text{OH})_{y+z}$. Iridium oxide can be formed by thermal growth, sputtering or anodic growth [12,13]. Use of anodic growth allows careful control of the degree of oxide formation and can be applied to iridium metal of any geometry, making it the most widely applicable technique. Activated iridium has been used

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successfully for neural stimulation in several animal models for acute and chronic implantation [14,15].

We recently reported a series of optical and electrochemical techniques for assessing the effective electrode area, charge capacity and charge density of platinum and doped conducting polymer modified electrodes [16]. This work showed significant variation from nominal to geometric electrode area. The electrochemically active area could also be measured by cyclic voltammetry of a redox species to give a linear or steady state diffusion area. The linear diffusion electroactive area provided information on the electrode roughness while the steady state diffusion area could assess the electrode geometry. The effective area was dependant on the measurement technique and was greatly affected by conducting polymer modification. The charge capacity and charge density of these electrodes also depended on measurement technique, raising questions on the best method for measuring these parameters. A detailed investigation of the effective electrode area, charge capacity and charge density of iridium and activated iridium electrodes hasn't been performed. This article investigates these properties to compare the *in vitro* behaviour of activated iridium with doped conducting polymers, and so provide a more rational choice of the best electrode materials for *in vivo* use.

2. Experimental

Hexaammineruthenium(III) chloride ($\text{Ru}(\text{NH}_3)_6\text{Cl}_3$), potassium ferricyanide (Sigma-Aldrich) and 99.0% di-sodium phosphate (Fluka) were used as received. Electrode arrays were 4 shank, 32 electrode (8 electrodes per shank), $413 \mu\text{m}^2$ nominal geometric area iridium electrodes with $200 \mu\text{m}$ pitch (Neuronexus Technologies – $A4 \times 8\text{-}5\text{mm-}200\text{-}200\text{-}413$). Electrodes were tested in a 3 electrode configuration on a CHI660B potentiostat with CHI684 multiplexer (CH Instruments) using a Ag/AgCl (3M KCl) as reference electrode and Pt wire as counter electrode. Individual microelectrodes were electrochemically activated and tested in a

solution of 0.3 M phosphate buffer in deionised water. Activation was achieved by applying a cathodic pulse of -0.8 V for 0.5 s and then an anodic pulse of 0.8 V for 0.5 s for 4 different number of repetition cycles (50, 125, 250 and 500). The electroactive areas were measured by addition of $5 \text{ mM Ru}(\text{NH}_3)_6^{3+}$. Test solutions were not degassed to better represent conditions *in vivo*. 2 probes were tested, 4 electrode sites at each number of activation cycles (Fig. 1) [17], leaving 16 unactivated iridium electrodes as controls.

Electrodes were imaged using a BX61 optical microscope (Olympus) and the 2 dimensional geometric area measured with ImageJ (Fig. 1). Scanning electron microscopy (SEM) images were captured on a JEOL 840A. Charge density measurements were performed using cyclic voltammetry over a range of 0.8 to -0.8 V vs Ag/AgCl at a scan rate of 100 mV s^{-1} . Electroactive area measurements were undertaken over a range of 0 to -0.5 V varying the scan rate from 10 mV s^{-1} to 1 V s^{-1} . Electrochemical modelling was achieved with DigiElch.

3. Results

The colour of each microelectrode was captured via optical microscopy after electrochemical activation and was relatively consistent depending on number of activation pulses (Fig. 1). The unactivated iridium appeared bright silver; after 50 activation pulses, a non-uniform brown colour appeared on the electrodes; after 125 pulses the entire electrode surface had become brown; after 250 pulses the electrode was completely dark blue; and after 500 pulses the electrode colour had become a pale blue.

The iridium electrodes were activated by applying repeated cathodic and anodic pulses of 0.5 s. The amperometric curve for both electrode polarities displayed sharp decaying peaks with maximum currents $\sim 100\text{-}150 \text{ nA}$ on the first pulse, increasing to $\sim 5\text{-}10 \text{ uA}$ on the 500th pulse (Fig. 2a). Integration of the current-time curve during electrode activation provided the total charge passed for electrode activation. Different features are seen on the total activation charge versus time curve (Fig. 2b). The total charge

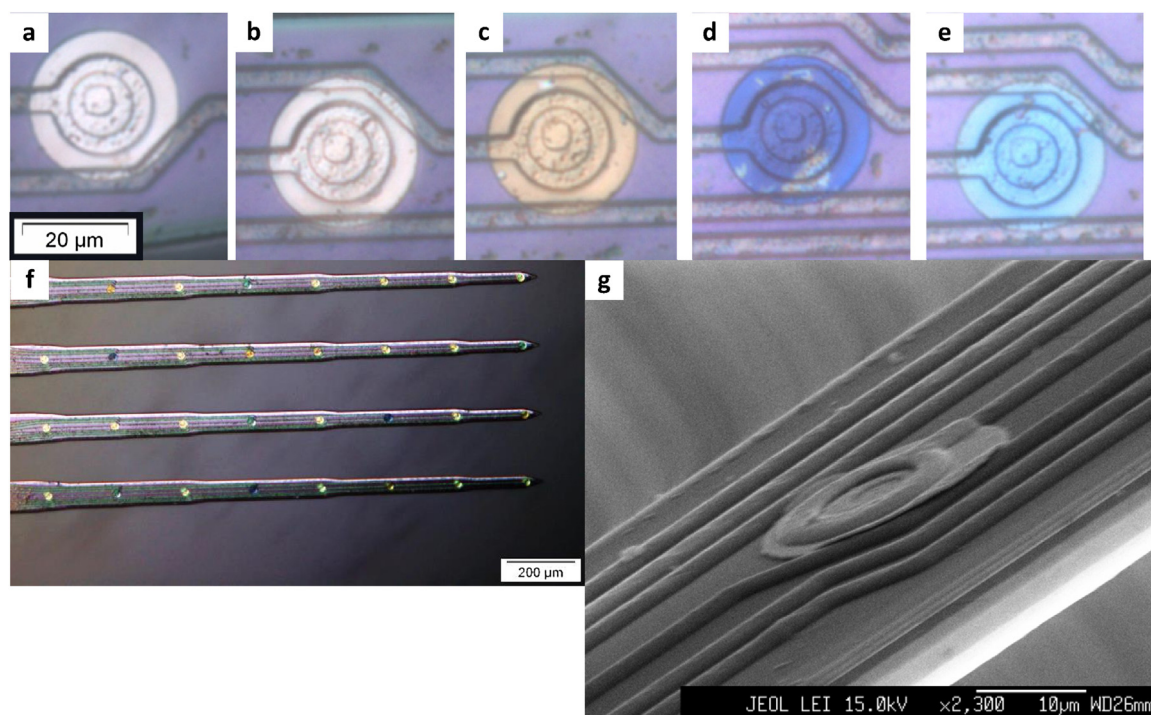


Fig. 1. Optical microscopy of iridium electrodes (a–e) after activation for 0, 50, 125, 250 and 500 pulses, (f) whole 32 channel array. (g) SEM image of an electrode and the connecting tracks.

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