



# A cost-effective fabrication of iridium oxide films as biocompatible electrostimulation electrodes for neural interface applications



Yong-Min Chen <sup>a</sup>, Tsai-Wei Chung <sup>a</sup>, Pu-Wei Wu <sup>a,\*</sup>, Po-Chun Chen <sup>b,\*</sup>

<sup>a</sup> Department of Materials Science and Engineering, National Chiao Tung University, Hsinchu 30010, Taiwan

<sup>b</sup> Department of Materials & Mineral Resources Engineering, National Taipei University of Technology, Taipei 10608, Taiwan

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## ABSTRACT

Electrostimulation medical devices for neural diseases require electroactive and biocompatible interface materials to transmit signals from electrodes to targeting tissues. Iridium oxide is an attractive ceramic material for neurostimulation electrodes due to its desirable stability and biocompatibility. In this study, we developed a cost effective and thickness controllable process to fabricate iridium oxide film by chemical bath deposition (CIROF). Surface morphology, crystallinity, roughness, hydrophilicity, and charge storage capacity as well as biocompatibility of the CIROFs with different thicknesses were analyzed. Accordingly, the CIROFs present rough and hydrophilic surfaces for cell attachment and good biocompatibility for cell viability. In addition, the charge storage capacity (CSC) of the as-deposited and the annealed CIROFs were 78.29 mC/cm<sup>2</sup> and 71.76 mC/cm<sup>2</sup>, respectively. Consequently, these results show advantages that make the CIROF a promising neurostimulation electrode material for implantable medical devices in neural systems.

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## 1. Introduction

The development of implantable neurostimulation electrodes in neural systems requires a new generation of electroactive and biocompatible interface materials that are able to transmit signals without any secondary effect related to charge transfer at the electrode interface [1,2]. The neurostimulation electrodes implanted in biological systems are limited to operate at a safe voltage to prevent damages in cellular media. Consequently, negligible impedance effect such as radical formation occurs during the implant operation. Accordingly, the neurostimulation electrodes used in the neural systems are expected to have desirable conductivities to keep cells viable and prevent unnecessary damages to surrounding tissues [3]. Therefore, the electrode materials used in the neural systems are commonly classified into two distinct categories depending upon charge transfer mechanism at the electrode interface; (1) capacitive charge injection materials: gold, titanium nitride, tantalum oxide, and (2) faradaic charge injection materials: platinum, platinum/iridium alloy, iridium oxide, and PEDOT [4–6]. In general, electrostimulation with faradaic

mechanism brings about better charge injection performance than that with capacitive mechanism. Among them, iridium oxide, a functional ceramic material, sufficiently provide necessary charges without damaging surrounding tissues for stimulation by electrochemical reduction and oxidation reactions at the electrode interface [1,7]. As a result, advantages such as high charge injection capability, desirable stability and requisite biocompatibility render iridium oxide becoming the most promising material for implantable neurostimulation electrodes.

Iridium oxide is typically formed from Ir metal by various techniques including, thermal decomposition (TIROF), reactive sputtering (SIROF), and electrochemical activation (AIROF) [2,8–10]. Alternatively, iridium oxide films can also be synthesized from iridium salt solution by electrodeposition (EIROF) [2,11–13]. However, some limitations on the techniques listed above have been reported and revealed. For instance, leakage issue has been pointed out for the sputtered iridium oxide electrode due to poor adhesion and denseness, which commonly occurs at the edge of the coating layer [14].

In contrast, chemical bath deposition of iridium oxide does not require heating and vacuum system compared to processes of reactive sputtering deposition and thermal decomposition [15]. Moreover, this process is able to overcome the aspect ratio limitation to the substrates. In addition, the other advantage of the

\* Corresponding authors.

E-mail address: [cpc@ntut.edu.tw](mailto:cpc@ntut.edu.tw) (P.-C. Chen).

chemical bath deposition process is it does not need conductive substrates which are required in electrodeposition, so it allows the formation of uniform and high-quality iridium oxide film not only onto conductive targeting substrates [16]. It is significant that each preparation method may yield a substantially different material such as impurity, oxygen content, and structure. For iridium oxide films, final redox states, charge storage capacities, and impedances at the interface are highly dependent on the preparation methods and the resulting structures. For these reasons, chemical bath deposition is an alternative solution to the iridium oxide film synthesis for implantable electrostimulation electrodes.

In this study, a cost effective and thickness controllable process by chemical bath deposition was carried out to fabricate functional ceramic of iridium oxide. The CIROFs were investigated and characterized its properties and performance at room temperature. The CIROFs were also tested for the cell viability to confirm its biocompatibility for further implantation applications. In addition, iridium oxide films have also been explored in other applications on other fields such as catalysis, electrochromic devices and ferroelectric memories [17–20]. Therefore, the development of the functional ceramic films shown here may also be significant in other areas.

## 2. Materials and methods

### 2.1. Preparation of coatings

The chemical deposition bath for growing iridium oxide film was prepared with a total volume of 9.7 mL. The plating bath contained 0.01 M of  $\text{Na}_3\text{IrCl}_6 \cdot x\text{H}_2\text{O}$  (Aldrich), 0.15 M of  $\text{NaClO}$  (SHOWA), 0.03 M of  $\text{NaOH}$  (Mallinckrodt), and 0.01 M of  $\text{NaNO}_2$  (SHOWA). A schematic diagram of the preparation for the plating bath and the deposition process is depicted in Fig. 1. First,  $\text{Na}_3\text{IrCl}_6 \cdot x\text{H}_2\text{O}$  was dissolved in DI water, forming a pale yellow solution.  $\text{NaNO}_{2(aq)}$ ,  $\text{NaOH}_{(aq)}$ , and  $\text{NaClO}_{(aq)}$  were dissolved in DI water separately, and then these three solutions were mixed together to become a transparent mixture. Once the transparent mixture was added to the  $\text{Ir}^{3+}$  precursor solution, the resulting bath turned to light yellow and the pH value of the solution was 12. At this stage, an ITO-coated glass over an area of  $1 \times 2 \text{ cm}^2$  was utilized as the substrate for chemical bath deposition, and it was rinsed with acetone and DI water to remove debris and contaminants prior to the chemical bath deposition. Finally, the ITO-coated glass substrate was immersed into the plating bath at 25 °C. Moreover, multilayer deposition was carried out to alter the film thickness following the same procedure described above. In this work, the goal is to prepare a 1  $\mu\text{m}$  CIROF and 1-layer, 3-layer, 7-layer, 11-layer, 15-layer, and 21-layer of CIROFs will be characterized. After finishing multilayer deposition and obtaining a 1  $\mu\text{m}$  CIROF, annealing was carried out in air environment at 450 °C for 2 h.

### 2.2. Surface characterization

Surface morphology and film thickness were observed under a field-emission scanning electron microscope (FE-SEM; JEOL-JSM-6700F). An atomic force microscope (AFM; Bruker Innova) was involved to determine the roughness of the films, and it was utilized for a  $3 \times 3 \mu\text{m}^2$  scanning area at tapping mode. The contact angle measurement (First Ten Angstroms PCS-1000B) was carried out to determine the hydrophilicity/hydrophobicity of the sample surface. The setup includes a camera, a light source, a power supply, and a micro-dropper which can drop 2  $\mu\text{l}$  of DI water on the sample surface. The shape of the droplet was captured and the contact angle can be calculated from the angle between the solid and a tangent aligned with the liquid at the point of contact with the

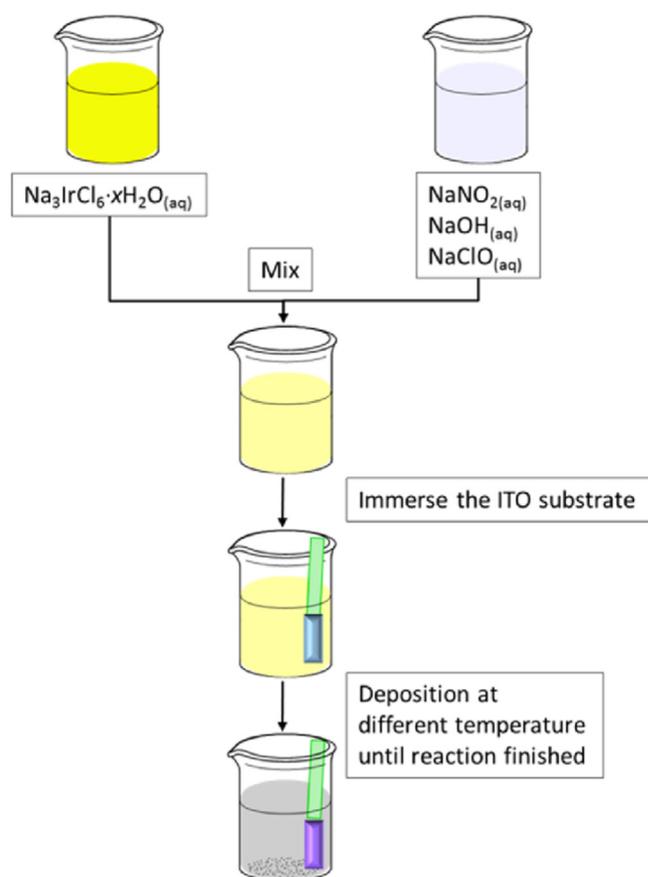


Fig. 1. A schematic flowchart of the chemical bath deposition of iridium oxide.

solid.

A high-resolution XRD (Bruker D2 Phaser) with 1.54 Å  $\text{Cu K}\alpha$  target was employed to identify the crystallinity of CIROF and the scan rate was  $0.05^\circ/\text{s}$  from 10 to  $90^\circ$ . An X-ray photoelectron spectrometer (XPS; Thermo Microlab 350) was used to identify the oxidation states of existing elements in the films. For the XPS analysis, additional Pt particles were sputtered on the film surface serving as a standard for correction with metallic Pt binding energy of  $4f_{7/2}$  at 71.2 eV.

### 2.3. Electrical properties measurement

Hall Effect was used to measure resistivity of the CIROFs by ECOPIA Hall effect measurement system (HMS-3000). The Hall Effect and van der Pauw method were used to determine resistivity of the 1  $\mu\text{m}$  as-deposited and the 1  $\mu\text{m}$  annealed CIROFs. The sample size was  $1 \times 1 \text{ cm}^2$ , and the input current was set as 1 mA with an applied magnetic field of 0.55 T.

Cyclic voltammetry (CV) was carried out in a phosphate buffered saline (PBS) electrolyte with the Jiehan potentiostat. The working electrodes were  $1 \times 1 \text{ cm}^2$  of the as-deposited and annealed CIROFs with thickness of 1  $\mu\text{m}$ . A platinum sheet was used as a counter-electrode, and an  $\text{Ag}/\text{AgCl}$  electrode as a reference electrode. Also, a Pt-coated ITO substrate was prepared and measured its electrochemical behavior as a control group. The charge storage capacity (CSC) was determined by integration of cathodic current in a potential window for iridium oxide between  $-0.6$ – $0.8 \text{ V}$  at  $50 \text{ mV}^{-1}$ . It has become common practice to characterize the stimulation electrodes by measuring the cathodic charge storage capacity (CSC). The CSC was determined by integration of the cathodic

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