

A Novel Technique for Increasing Charge Injection Capacity of Neural Electrodes for Efficacious and Safe Neural Stimulation*

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Abstract— Neural prostheses require chronically implanted small area penetrating electrode arrays that can stimulate and record neural activity. The fundamental requirement of neural electrodes is to have low interface impedance and large charge injection capacity (CIC). To achieve this fundamental requirement, we developed a novel technique to modify the surface of the Utah Electrode Array (UEA) to increase the real surface area without changing the geometrical surface area. It was coated on modified and unmodified (control) UEAs and electrochemical characterization such as impedance and CIC was measured and compared. The surface modified electrode impedance and CIC was ~ 188 Ohm and ~ 24 mC/cm² respectively. Increasing the real surface area of electrodes decreases the impedance by 1000 times and increases the CIC by 80 times compared to the control samples. The CIC of modified UEA was significantly higher than of any material reported in the literature, higher than sputtered iridium oxide (4 mC/cm²) or PEDOT (15 mC/cm²).

I. INTRODUCTION

Functional electrical stimulation of nerve tissue and recording of neural electrical activity are the foundation of emerging prostheses and treatments for spinal cord injury, stroke, sensory deficits, bladder prostheses, retinal and cortical visual prostheses, epilepsy, and other neurological disorders [1]. The recent demonstration of wilful computer cursor movement by a tetraplegic patient offer hope for neural prostheses [2]. The efficacy of neural devices is ultimately determined by the quality of the neural-electrode interface, which in turn depends on the electrode characteristics. The ideal electrode used as a stimulating or recording neural activity must satisfy requirements such as (1) biocompatibility: should not induce a toxic or necrotic response in the targeted tissue, (2) mechanically robust: should be robust to withstand insertion forces, (3) efficacious: sufficient charge can be injected to elicit tissue response, and (4) safety: during the implant duration while the electrodes are active (recording or stimulation), the electrode should not generate any reaction which are toxic or lead to premature failure of electrode/device [3].

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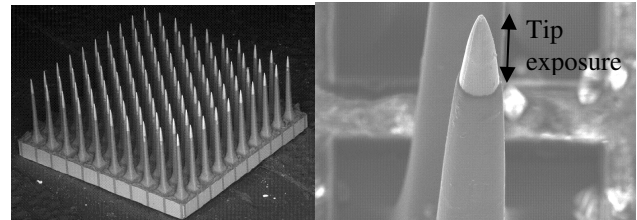


Figure 1. SEM micrograph of the Utah Electrode Array (UEA) showing the entire array (left) and a view of one electrode at high magnification to illustrate the tip exposure (right). The UEA is encapsulated by an insulating parylene-C layer with exception of the tip ($\sim 50\mu\text{m}$) of the electrode which forms the active site for stimulation or recording of neural signals.

To record or stimulate single unit activity, the electrodes need to have high selectivity and sensitivity. The selectivity is defined as the ability of an electrode to activate a small population of neurons without activating neighboring populations. The small surface area of active sites of the electrode improves the spatial resolution and selectivity. However, as the area of active sites decreases, the electrode impedance increases, which in turn affects the recording/stimulating characteristics (sensitivity). Thus, there is a design trade-off between selectivity and sensitivity. To overcome this trade-off, electrode materials with higher charge injection capacity (CIC) are desired to allow smaller electrodes. Small electrodes achieve higher stimulation current density while operating within safe voltage limits that avoid gas evolution by electrolysis or electrochemical reactions. Furthermore, higher CIC is needed to lower the potential required for stimulation, which could reduce injury at the stimulation site and also affect the longevity of the electrode/device. In the literature, many materials have been studied of which the most popular ones are platinum, iridium oxide, poly(3,4- ethylenedioxythiophene) (PEDOT) and carbon nanotubes (CNT). As seen in Table 1, among these materials, the highest CIC reported till date is of PEDOT (15 mC/cm²) [4].

In this paper, we have devised a novel technique to overcome the above stated design trade-off. Our approach is to generate large real surface area (RSA) without increasing the geometrical surface area (GSA) of the active sites of the electrodes. Large area of the electrode can be achieved by tailoring the electrode surface morphology. Making the electrode rough increases the RSA without increasing the GSA of the electrode. In this paper, we have modified the electrode surface and then coated the electrode with platinum (Pt) metal to yield CIC of 24 mC/cm² which is 80 times higher than unmodified electrode coated with Pt. The modified UEA CIC is significantly higher than PEDOT.

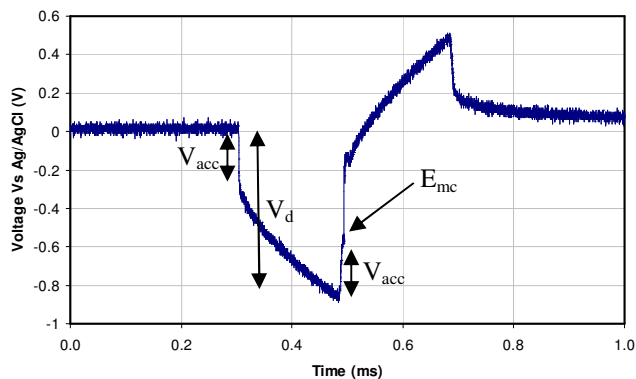


Figure 2. Voltage transient of an electrode of the UEA in which biphasic, symmetrical current pulse was passed at 50 Hz. The figure highlights maximum cathodic potential ($E_{mc} = -0.5$ V) during a pulse.

II. METHODOLOGY

A. Surface Modification

A detail description of the fabrication of the Utah Electrode Array (UEA) is given elsewhere [5]. The tips of the UEA were selectively roughened at different RIE power in a capacitive coupled plasma of SF_6 (Oxford Plasmalab 80 plus) using aluminum foil as a mask. The flow rate of SF_6 was 26 sccm while RIE power was varied from 100-300 W [6]. All the samples were etched for 20 min. Roughness was inspected in the scanning electron microscopy (SEM), using an FEI Nova NanoSEM microscope. A metal stack comprising of Ti/Pt were selectively DC sputtered on the tip of the electrodes by using aluminum foil as a mask. Ti acts as an adhesive layer and was deposited using DC sputtering. The base pressure of the sputter was less than 10^{-7} Torr. The Ti layer was sputtered in Ar ambient at a chamber pressure of 10 mTorr with Ar flowing at 150 sccm (standard cubic centimeters per minute) and sputtering power of 90 W for 5 min. The sputtering parameters were optimized to achieve low stress Ti film. Ti target was 99.6% pure, 3 inch in diameter and 0.125 inches in thickness (Kurt J. Lesker). The deposition rate of Ti was 10 nm/min. Pt films were dc sputtered. The Pt cathode was 99.5% pure, 3 inch in diameter and 0.125 inches in thickness (Kurt J Lesker, Pittsburgh, PA). A process pressure of 11 mTorr was achieved using the throttle valve and an Ar gas flow rate of 150 sccm. Sputtering was done at 90 W for 10 min. The deposition rate of Pt was 20 nm/min. The film thicknesses were measured with a Tencor P-10 profilometer on a silicon witness wafer masked to yield a step. The surface morphology and tip exposure of Ti/Pt coated UEAs were examined by SEM.

B. Electrochemical Characterization

Cyclic voltammetry (CV) and potential transient responses during current pulsing were measured in a three-electrode cell consisting of Ag/AgCl as a reference electrode, a large area Pt wire as a counter electrode and the UEA electrodes as working electrodes. All potentials were

measured with respect to the reference electrode. CV was acquired in a physiological phosphate buffer saline (PBS) solution at room temperature in a commercial electrochemical test system (Gamry Instruments (PC4 potentiostat), Warminster, PA). The cyclic voltammograms were recorded at a 50 mV/s sweep rate between potential limits of -0.6 V and 0.8 V (water window), beginning at the open circuit potential and sweeping in the positive direction first. The water window was considered if the E_{mc} becomes less than -0.6 V [7]. The charge storage capacity (CSC) was calculated using the following equation

$$CSC = \frac{1}{\nu A} \int_{E_c}^{E_a} |i| dE \quad (1)$$

where E is the electrode potential (V versus SSE), i is the measured current (A), E_a and E_c are the anodic and cathodic potential limits (V), respectively, A is the GSA of the exposed tip (cm^2) and ν is the scan rate. In this work, the UEAs had a tip exposure of $50 \mu m$ ($GSA = 2 \times 10^{-5} cm^2$).

Electrochemical impedance spectroscopy (EIS) was performed in the Gamry system which was used to record the CV. Impedance (Z) was measured for frequencies from 1 Hz to 100 kHz by applying a sinusoidal signal having amplitude of 10 mV.

For CIC measurements, current pulsing was performed with a STG 2008 stimulator generator (Multi-Channel Systems MCS GmbH, Germany). Current pulses were delivered as charge-balanced biphasic pairs, cathodal first, with equal times and current amplitude for each phase. The pulse frequency was kept constant at 50 Hz, allowing ~ 19 ms time period between pulses. The cathodal pulse width of 0.2 was used to compare the CIC of modified and unmodified UEAs. The potential transient was recorded with an oscilloscope and the maximum negative potential excursion (E_{mc}) was calculated by subtracting the access voltage (V_{acc}), associated with ohmic resistance, from the maximum negative voltage in the transient. E_{mc} is also equal to the potential immediately after the end of the cathodic pulse when V_{acc} is zero, as illustrated in fig. 2. V_d denotes the driving voltage, which is defined as the maximum voltage required to deliver the current pulse. The CIC of modified and unmodified UEA was calculated by multiplying stimulation current and pulse width at which the potential (E_{mc}) reaches water reduction potential (-0.6 V) divided by the GSA.

III. RESULTS

Fig. 3 shows the SEM micrographs of the electrodes of the UEA which were plasma etched at different RIE power. Notice that as the RIE power increases electrode roughness also increases. However, increasing the RIE power makes the electrodes fragile. The UEA needs to withstand the insertion forces and hence need to be mechanically robust. To find the optimum roughness and yet mechanical robustness, the UEA were repeatedly inserted into 2% Agarose. It was found that all the modified electrodes were able to withstand Agarose insertion test except the electrodes etched at 300 W RIE, which broke from the tips. Hence,

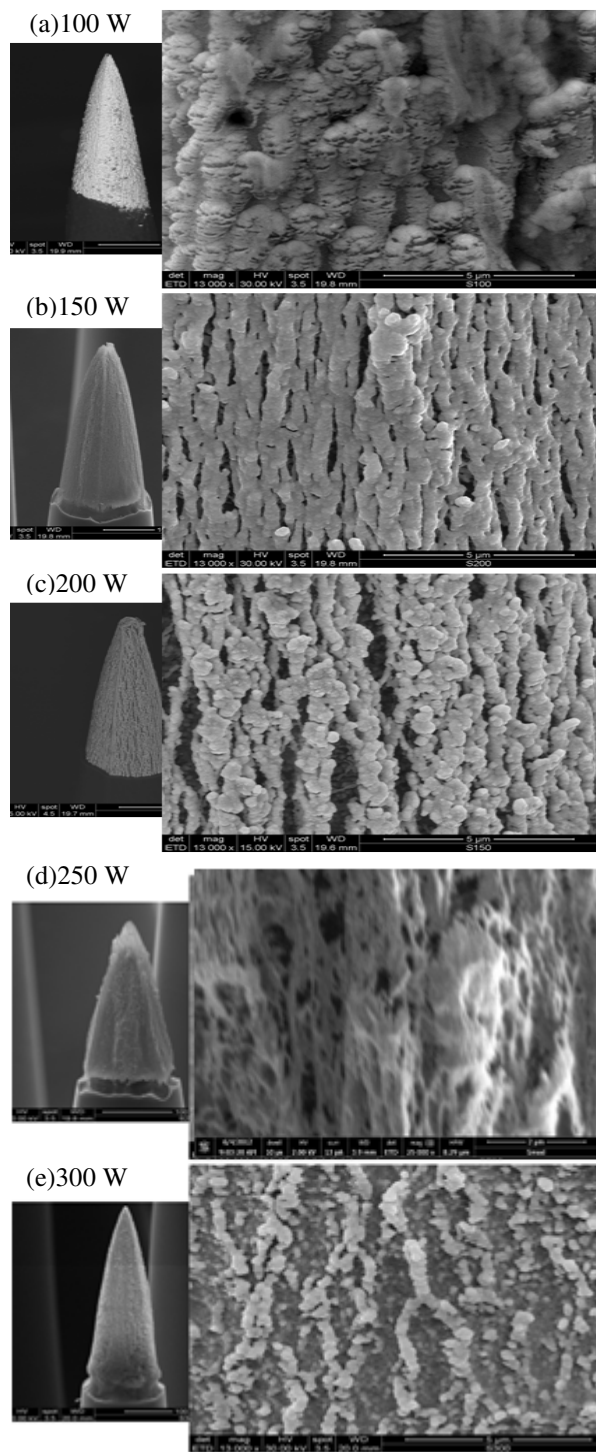


Figure 3. SEM micrographs of SF₆ etched UEA tips at RIE power (a) 100 W, (b) 150 W, (c) 200 W, (d) 250 W, and (e) 300 W. The insets shows the zoom-in micrographs of the surface to illustrate microstructure of the surface.

electrochemical characterization was done for electrodes etched at 250 W as at this power the electrodes have maximum roughness, lowest impedance and were mechanically robust to withstand the insertion forces. The average impedance (at 1 kHz) of 96 electrodes (modified) is 188 Ω with 24 Ω standard deviation while that of unmodified

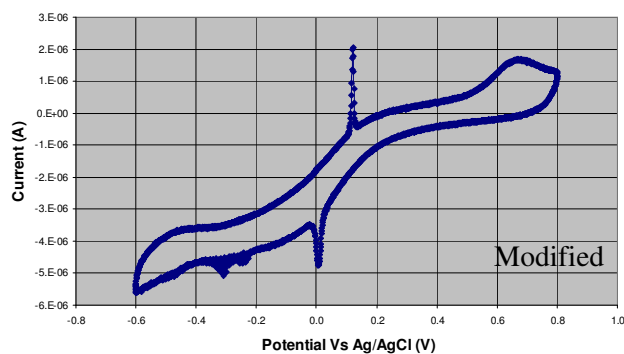


Figure 4. Cyclic voltammograms of the surface modified Pt coated UEA

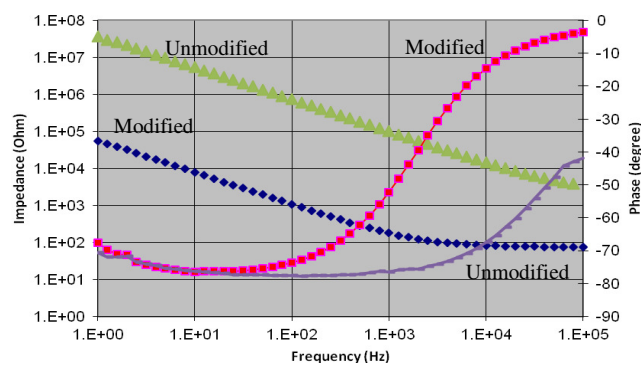


Figure 5. Typical Bode plot presents the electrode impedance as a function of frequency of the Pt coated unmodified/control and surface modified electrodes of the UEA. At all frequency measured, impedance of the surface modified electrode is significantly lower than that of control samples coated with same metal, Pt. At higher frequencies (greater than 100 Hz) the modified surface have more resistive component than capacitive.

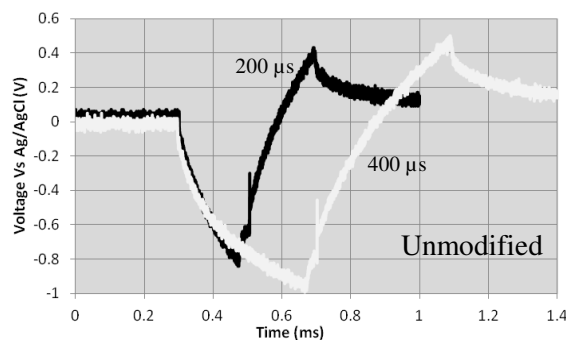


Figure 6. Voltage transient for unmodified Pt coated UEA in response to a 30 μA current pulses of 200 and 400 μs duration. It can be seen in the graph that at 200 μs the E_{mc} is at -0.6V but at 400 μs it decreases to -0.8 V which is outside the water window.

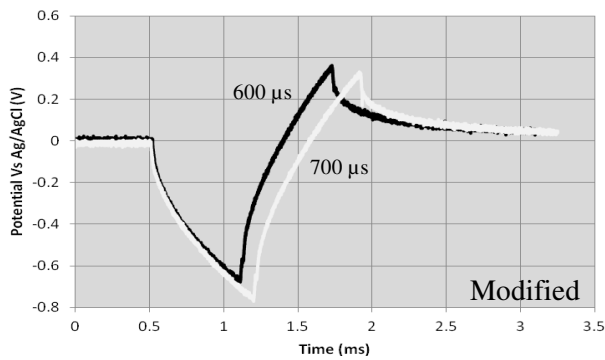


Figure 7. Voltage transient for surface modified Pt coated UEA in response to a 800 μ A current pulses of 600 and 700 μ s duration. It can be seen in the graph that at 600 μ s the E_{mc} is at -0.6V but at 700 μ s it decreases.

TABLE I. CIC OF ELETRODE MATERIALS FOR STIMULATION

Materials	Mechanism	CIC (mC/cm^2)
Pt [8]	Capacitive	0.3
Activated iridium oxide [4, 9]	Faradaic	4
Sputtered iridium oxide [8]	Faradaic	4
TiN [9]	Capacitive	0.55
Ta ₂ O ₅ [4]	Capacitive	0.5
PEDOT [4]	Faradaic	15
CNT [4]	Capacitive	1.6
Surface modified Pt	Depends on material	24

is 159 $\text{k}\Omega$ with standard deviation of 64 $\text{k}\Omega$. Fig. 4 shows a representative cyclic voltammogram of modified electrode surface Pt coated UEA. The average CSC calculated from equation 1 is 3000 mC/cm^2 while that of unmodified Pt coated UEA is 4.4 mC/cm^2 . Due to the increase in surface roughness, CSC increased 1000 times. Fig. 5 shows Bode plot comparison of surface modified and unmodified Pt coated UEA. It can be seen that in all frequency the impedance of surface modified UEA is less than unmodified UEA. The unmodified UEA has capacitive behavior however modified UEA has more resistive component at higher frequencies. Fig. 6 and 7 shows the potential transient of the unmodified and modified UEA, respectively. The CIC of unmodified UEA is 0.3 mC/cm^2 while that of modified UEA is 24 mC/cm^2 . Due to increase in RSA / roughness, CIC of Pt coated UEA increases by 80 times. The modified UEA's CIC is highest ever reported for any type of material; even higher than that of PEDOT with 15 mC/cm^2 or sputtered iridium oxide with 4 mC/cm^2 [4, 8]. Table 1 summarizes the CIC of various materials in literature. It will be interesting to study and evaluate surface modified UEA coated with sputtered iridium oxide for stimulating and recording neural signals.

IV. CONCLUSION

It is envisioned that the presented novel surface modification technique would improve selectivity, sensitivity and precision during physiological experiments. Smaller tip exposure of the UEA would lead to higher selectivity without compromising on sensitivity. Due to the small area and low impedance electrodes, signal to noise ratio of neural signal is expected to increase which will help researchers to interpret the data more reliably. Ultimately, this method will help in chronic application by increasing the longevity of the neural device. There is a need for extensive *in vivo* evaluation of stimulation characteristics of surface modified UEA. Even higher RIE power (> 250 W) etched UEA needs to be evaluated *in vivo* as there are strategies to reduce the insertion force by 40% which ultimately would relax the mechanical properties of the surface modified UEA [10]. Furthermore, we are depositing sputtered iridium oxide on the modified UEAs to evaluate the electrochemical characteristics for neural stimulation and recording neural signal.

DISCLAIMER

F Solzbacher has a financial interest in the company Blackrock Microsystems, which develops and produces implantable neural interfaces, and electrophysiology equipments and software.

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