

Diffusion-bonded Electrodes for Chronic Neural Stimulation

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Abstract— We report a novel method to fabricate chronic neural interfaces with the intent to combine the reliability and lifetime of bulk metal electrodes, with the miniaturization and mechanical flexibility of thin-film polymer microelectrode arrays. 10 μm thick platinum discs were laser cut from a foil into the shape of individual electrodes, and coated with gold on the backside. The discs were bonded to a microelectrode array with gold bond pads via gold-gold inter-diffusion using a flip-chip bonder. Electrode bonding and adhesion was characterized using mechanical shear testing and electrical testing. Electrode performance was characterized *in vitro* using electrochemical impedance spectroscopy and cyclic voltammetry. Biphasic electrical pulsing experiments were conducted on the bonded electrodes to study degradation of the electrode; the preliminary results show that the electrodes can withstand at least 4,900 million pulses with no adverse electrochemical or visual degradation. Overall, this is a promising new method for fabricating chronic neural electrodes for stimulation or recording that combines the reliability of commercial bulk electrodes with the miniaturization and versatility of microfabricated technologies.

I. INTRODUCTION

The use of electrical stimulation and recording to interact with neural tissue is finding growing application in understanding the fundamentals of neuroscience [1]. Ultimately, this understanding will lead to the development of clinical treatments for neurological diseases such as Parkinson's, epilepsy, loss of vision or hearing, spinal cord injury, and neuropsychiatric disorders [2]. Although there are several approved clinical neural stimulation treatments, further advances in device technology could greatly improve such therapies [3].

Most clinical neural interfaces today utilize bulk platinum electrodes that are welded to wires. Though this approach is well characterized, this manufacturing technique significantly limits both the number of electrodes and the density. To achieve higher numbers of electrodes, the size of the implant becomes unacceptably large. As a result, most implantable, clinical, neural interfaces have just a few electrodes placed across a relatively large area. In certain treatments such as deep brain stimulation (DBS), cochlear implants, and spinal cord stimulation, treatment outcomes may be improved by the ability to make smaller devices with larger numbers of more densely-packed electrodes that can

safely deliver large amount of charge in a controlled manner [4,5].

The fabrication of microelectrode arrays using semiconductor process technology promises to miniaturize neural interfaces by decreasing pitch and increasing the number of electrode sites [6]. Microelectrode arrays have been successful in many acute neural interface applications. However, there are a few hurdles before this technology can be widely adopted in clinical practice. Specifically, a microelectrode array must 1) not elicit a cytotoxic response in the body 2) not corrode or degrade over time, and 3) maintain performance over the lifetime of the patient. Significant strides have been made in recent years to overcome these hurdles [7-10].

A significant unmet challenge for microelectrode-based neural interfaces is the ability to maintain electrode material performance over chronic, multi-year lifetimes. In most microfabricated neural interface technologies, electrodes consist of thin-film metals up to 1 μm in thickness. Depending on the combination of electrode size, electrical pulsing parameters, and array design, many of these thin-film materials degrade after limited electrical pulsing [11-13]. Even for moderate charge densities, the thin-film electrodes cannot sustain the same amount of charge injection as bulk electrodes over their lifetime. Standard microfabrication processes, such as physical vapor deposition or atomic layer deposition, do not easily permit the deposition of films of thicknesses much greater than 1 μm . In comparison, the bulk platinum electrodes used commercially today are 10-100 μm thick. There is a need to fabricate microelectrode technologies with greater thicknesses.

A method of increasing electrode performance and lifetime is to reduce the charge density by increasing the effective surface area or by roughening the electrode material surface. This is done today by coating electrodes with a highly-conductive material (e.g. poly(3,4-ethylenedioxythiophene) PEDOT) or electroplating platinum [14]. These materials can improve performance at higher charge densities by decreasing electrode impedances and material corrosion, but are known to have mechanical stability concerns over time. It would be advantageous to create high-surface area electrodes with enhanced lifetime and stability.

There have been prior efforts to create bulk electrodes with three dimensional microstructures to increase surface area and charge injection capacity. An example of this is a bump-bonded gold electrode for electrochemical detection of contaminants in water [15]. Impression molding was performed to enhance the electrode surface area [16]. For neural stimulation and recording application, we are

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interested in extending such techniques to platinum electrodes.

This work presents a novel method of fabricating microelectrode arrays with diffusion-bonded, bulk platinum electrode material that is designed to be electrochemically stable. This method can be extended to a wafer-scale; thus, the benefits of micro-fabrication are not lost. We present the characterization results of bulk platinum (Bulk-Pt) electrodes as they relate to the performance required for chronically stimulating electrodes for neural interface.

II. FABRICATION

Fig. 1 shows the microfabrication process for the diffusion-bonded platinum (Bulk-Pt) electrode arrays. A silicon wafer with silicon nitride passivation was used as the starting substrate (Fig. 1A). A trace metal layer consisting of Ti/Au (20/500 nm) was sputter-deposited and patterned using contact lithography and wet metal etching (Fig. 1B). Separately, a 100% platinum foil of 10 μm thickness was laser cut to produce circular electrodes, 600 μm in diameter. These bulk electrode discs were sputter-coated on the backside with a Ti/Au (20/500 nm) (Fig. 1C), and bonded to the trace metal layer using flip-chip technology (Finetech Lambda, Gilbert, AZ) (Fig. 1D). Each Ti/Au-coated platinum bulk electrode disc was aligned to the bond pad of the gold trace layer with the Ti/Au-coated side facing the gold bond pad. Then, a force of 200N was applied through the tool head with a roughened surface to impart texture to the Pt disc. Simultaneously, the tool head's temperature was raised to 150 $^{\circ}\text{C}$ and the stage's temperature was raised to 330 $^{\circ}\text{C}$ for 5 minutes to promote gold-to-gold inter-diffusion thereby achieving the desired bond. A top insulating layer of 5 μm polyimide was spin-coated on the substrate and cured (Fig. 1E). Finally, an electrode opening, 400 μm in diameter, was etched above the Bulk-Pt electrode using O_2 plasma (Fig. 1F). It is important to point out that in the ultimate application of this technology, the entire microelectrode array would be flexible, and hence a bottom polyimide layer would be used so that the final device can be removed from the silicon carrier wafer, as our group has demonstrated in the past [7].

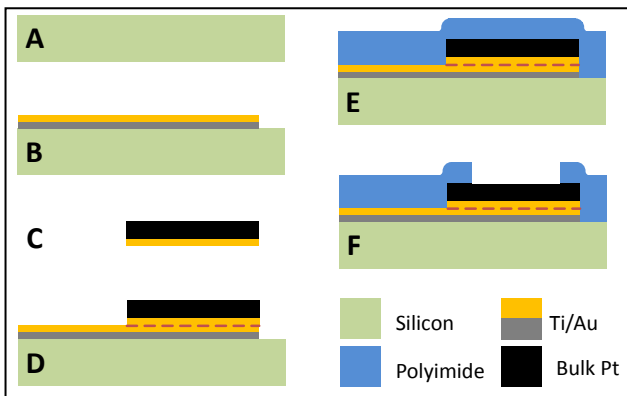


Figure 1. Process for diffusion bonding Bulk-Pt discs onto microelectrode arrays (Note: Images not drawn to scale)

III. IN-VITRO CHARACTERIZATION

A. Bond Adhesion Characterization

After diffusion bonding of the Bulk-Pt electrodes, the bond was characterized electrically by measuring the resistance between the top of the Bulk-Pt electrode and the top of the connector pad. This test was performed to quantify contact resistance due to the bond interface. The bonds were characterized mechanically using a shear testing module on an F&K Delvotec semi-automatic wire-bonder.

B. Electrochemical Characterization

Electrochemical impedance spectroscopy (EIS) was performed in order to quantify the performance of the electrode material. EIS measurements were made at frequencies from 100 kHz to 1 Hz with a 10 mV amplitude sinusoidal signal.

All electrochemical data was acquired at room temperature in phosphate-buffered saline (PBS) using a 3-electrode cell in a commercial electrochemical test system (PAR VersaSTAT 4 potentiostat, Oak Ridge, TN). A silver-silver chloride (Ag/AgCl) electrode was used as a reference electrode and a large surface area platinum wire was used as a counter electrode. All potentials were measured with respect to the Ag/AgCl reference electrode. Cyclic Voltammograms (CVs) were collected at a 100 mV/s scan rate over the potential range of -600 mV to +800 mV. Cyclic voltammetry (CV) was performed to determine the charge storage capacity (CSC).

To characterize the stability of the electrode material, the Bulk-Pt electrodes were pulsed continuously with a biphasic, cathodic-first, charge-balanced pulse (1) with an amplitude of 1mA for a 100 μs pulse width for 900 million pulses and then (2) with an amplitude of 2mA for a 100 μs pulse width and 4,000 million pulses.

IV. RESULTS AND ANALYSIS

A. Optical imaging

Fig. 2 shows the diffusion bonded Bulk-Pt electrodes in comparison to traditional thin-film platinum electrodes (TF-Pt). It is important to consider that both electrode metals had a diameter of 600 μm as deposited, however, only a 400 μm diameter of this platinum was exposed by plasma etching of the top insulating polyimide layer. Fringes are visible in the image of the Bulk-Pt electrode due to the varying polyimide thickness around the electrode perimeter. The thin-film platinum electrode chips were fabricated in a process similar to the Bulk-Pt electrodes, however, the electrode material was deposited using physical vapor deposition (sputtering) and was photolithographically patterned on the underlying trace metal using lift-off.

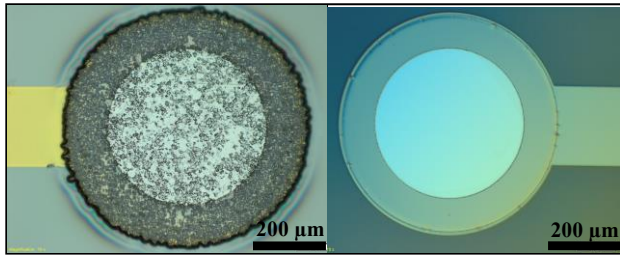


Figure 2. Optical microscope images of diffusion bonded Bulk-Pt electrode (Left), and TF-Pt electrode (Right). Note: the electrode openings in the top polyimide layer are $\sim 400 \mu\text{m}$ in diameter

B. Surface profile

The heights of the features of the Bulk-Pt electrode chips were characterized using a Dektak stylus profiler. The trace metal layer was $0.58 \mu\text{m}$ thick, and the roughened Bulk-Pt thickness ranged from $11\text{-}12 \mu\text{m}$.

C. Bond characterization

Electrical measurements were performed on a probe station to characterize the electrical contact of the Bulk-Pt electrode to the thin-film trace metal underneath. Table I compares the average electrical resistances measured between the bond pad and the connector pad, before and after bonding the Bulk platinum electrodes.

TABLE I. ELECTRICAL CHARACTERIZATION OF BOND ADHESION

Measurement location	Resistance (Ω)	n
Connector pad to electrode pad, no electrode	8.3	3
Connector pad to top of bulk-Pt electrode	9.2	6

The mechanical adhesion of the Bulk-Pt electrode on the trace metal layer was characterized using a shear test performed on an F&K Delvotec wire-bonder. The average peak shear force was 794.7 cN ($n=3$). Fig. 3 shows an optical microscope image of a Bulk-Pt electrode after shear test. A top layer of the platinum film sheared off, but there was no failure at the bond interface between the bulk electrode and the trace metal layer.

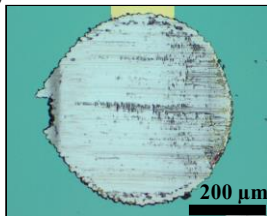


Figure 3. Optical microscope image of Bulk-Pt electrode after shear test.

D. Electrochemical Testing

Electrochemical impedance spectroscopy (EIS) was performed on the electrodes *in vitro* to record their impedances prior to long-term pulsing experiments. Lower impedance is desired for neural interfaces to allow for a reduced supply voltage and lengthened battery lifetime. Table II summarizes the average impedance and charge storage capacity of the Bulk-Pt and TF-Pt electrodes for comparison.

TABLE II. AVERAGE IMPEDANCE AND CHARGE STORAGE CAPACITY COMPARISON BEFORE ELECTRICAL PULSING

Electrode material	Average impedance at $f = 1\text{kHz}$ ($\text{k}\Omega$)	Average charge storage capacity (mC/cm^2)	n
TF-Pt	2.463	1.891	4
Bulk-Pt	2.013	1.339	6

The Bulk-Pt electrodes were inspected after applying 900 million pulses at 1mA amplitude and $100\mu\text{s}$ pulse width, and no visual changes were observed in the electrode surface, as shown in Fig. 4. The amplitude was increased to 2mA to accelerate the experiments. After an additional 4,000 million pulses at increased amplitude, no visual changes were observed on the electrode surface, except a sign of moisture leakage on the trace. These electrical tests are on-going.

The TF-Pt electrodes showed a few black pits across the electrode surface after 900 million pulses at 1mA amplitude and $100\mu\text{s}$ pulse width. After an additional 4,000 million pulses at 2mA were applied, the electrode surface was clearly covered with a layer of black/brown particles (Fig. 5), which is the sign of corrosion.

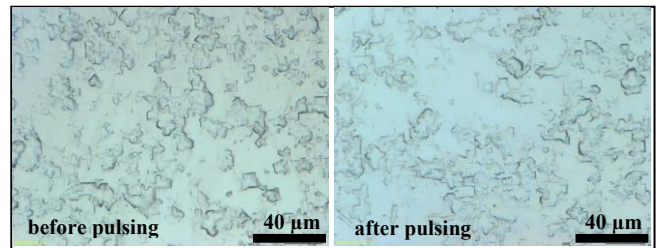


Figure 4. Optical microscope image of the Bulk-Pt electrode before (left) and after (right) electrical pulsing of 900 million pulses at an amplitude of 1mA for a $100\mu\text{s}$ pulse width. The images were not taken at the exact same location before and after pulsing; however, overall, there were no visual changes observed across the electrode surface. The texture on the electrode corresponds to the roughness created during the bond process to increase electrode surface area.

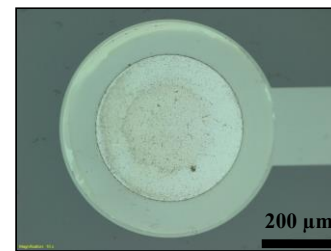


Figure 5. Optical microscope image of the TF-Pt electrode after 900 million pulses at 1mA amplitude and $100\mu\text{s}$ pulse width plus 4,000 million pulses at 2mA amplitude and $100\mu\text{s}$ pulse width.

V. DISCUSSION

Microfabricated, thin-film electrode arrays have limitations in neural stimulation applications requiring high charge densities due to their limited ability to withstand corrosion [10,12]. In order to address these limitations, we have demonstrated a process of bonding bulk electrodes onto a thin-film trace metal layer using diffusion bonding. This technique can be extended to a variety of different electrode materials including metals and non-metals as long as a sufficient diffusion gradient can be created between the electrode material and the underlying trace metal layer.

Bond quality was characterized electrically by comparing the resistances on the trace metal layer between the connector pad and bond pad, before and after bonding the bulk electrodes (Fig. 6). The addition of the bulk platinum led to an insignificant increase in the resistance which indicates high-quality electrical contact between the diffusion-bonded electrode and the underlying trace metal layer. It is important to note that resistance increase we observed is negligible compared with the typical range of 100-1000s ohm trace resistances of microelectrode arrays.

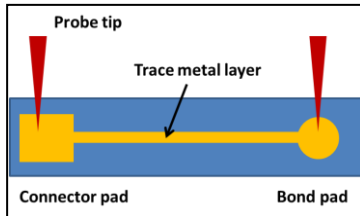


Figure 6. Electrical testing of Bulk-Pt electrode adhesion to trace metal layer.

Shear test demonstrated that the adhesion bond force was able to withstand a shear test without failing at the bond interface. This test is similar to ball and die shear tests performed in the microelectronics packaging industry to quantify bond strength.

After fabrication, the individual electrodes were characterized using electrochemical impedance spectroscopy. The impedances and charge storage capacities were similar to the results we have obtained with utilizing thin-film electrodes [3, 6-8].

The Bulk-Pt electrodes have been pulsed in the order of a billion cycles without obvious visual changes. This result compares favorably with pulsing experiments on TF-Pt electrodes that have a limited lifetime due to finite corrosion of the flat metal films [3]. Pulsing experiments are on-going to qualitatively and quantitatively discern changes in the electrode performance over time.

VI. CONCLUSION

We have demonstrated a novel method to combine the advantages in lifetime and performance of bulk platinum electrodes, with the versatility and miniaturization of thin-film microelectrode arrays. Preliminary lifetime testing shows no visible degradation of the electrode material after 4,900 million electrical pulses with moderate charge density. In future work, we intend to demonstrate a multi-electrode flexible array with bulk electrodes using the fabrication process we have outlined in this work.

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