ATOMIC LAYER DEPOSITED Al_2O_3 AND PARYLENE C DUAL-LAYER ENCAPSULATION FOR BIOMEDICAL IMPLANTABLE DEVICES

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ABSTRACT

We present a novel bi-layer encapsulation method combining atomic layer deposited Al_2O_3 and Parylene C to extend the lifetime of biomedical implantable devices. The atomic layer deposited alumina and Parylene C bi-layer coated interdigitated electrodes had leakage current of about 20 pA and impedance of 3.5 MΩ after 260 days of soaking in phosphate buffered solution at 67 °C (roughly 6 years of equivalent lifetime at 37 °C). Also 5 VDC continuous bias had no measurable effect on the lifetime of bi-layer coating while it shortened the lifetime of Parylene coating by 75%. The long-term low leakage current and high insulation performance under continuous bias demonstrated its suitability for encapsulation of chronic implantable devices.

KEYWORDS

Atomic layer deposited Al_2O_3, Parylene, electrochemical impedance spectroscopy, encapsulation of biomedical devices, accelerated lifetime testing

INTRODUCTION

There has be a continuous strong interest on developing biomedical implantable devices for research and clinic applications, such as cochlear implants[1], diaphragm pacing systems[2] and deep brain stimulators for treating diseases such as hearing loss, respiratory failure, and Parkinson's[3]. Implantable systems for long-term clinic trials require chronic implantations able to perform their intended functionalities for years or decades, in order to reduce surgical risks and generate levels of efficacy that justifies the risks associated with the implantation. In order to perform its intended use, the device has to be isolated from the physiological environment. The encapsulation of implantable device is critical to its functionality, stability and longevity. Traditionally, lids and cans based hermetic coatings have been used. However, they usually take more space compared with thin-film based encapsulation, especially for micro medical systems. Another challenge for hermetic encapsulation is feedthroughs. Therefore, thin-film based encapsulation has been preferred for implantable devices over hermetic encapsulation. Thin-film encapsulation has to be biocompatible, conformal, highly resistive, and have a low dielectric constant[4] and low water vapor transmission rate (WVTR). Parylene C[5-7] has been widely used as encapsulation material for different kinds of implantable devices, based on its low water absorption rate of 0.1% for 24 hours[8], low dielectric constant of 3.15 at 60 Hz[8], USP Class VI biocompatibility, and chemical inertness. Parylene C is also an excellent ion barrier[9], which is very important for implantable devices exposed in physiological environment. Failure of Parylene C encapsulation has been reported[10] due to moisture diffusion and interface contamination. In order to further improve the lifetime of implantable devices, moisture has to be isolated from the interface between the coating layer and the device itself. This will prevent moisture from condensing around interface contaminants.

Atomic layer deposited (ALD) Al_2O_3 has been demonstrated as an excellent moisture barrier[11, 12] with water vapor transmission rate (WVTR) of about 10^{-6} $g/m^2 \cdot day$, for preventing degradation of extremely moisture-sensitive organic light emitting diodes (OLEDs). Compared with films generated by other deposition techniques such as sputtered Al_2O_3, ALD Al_2O_3 is superior as a moisture barrier [11, 13] because the film is highly conformal and pin-hole free. Liquid water is known to corrode Al_2O_3[14]; therefore, Al_2O_3 film alone is not suitable for encapsulation of biomedical implants directly exposed to physiological environment. The idea of combining Al_2O_3 and Parylene is based on the concept that Al_2O_3 works as an inner moisture barrier and Parylene C as an external barrier to ions. Also, Parylene C prevents contact of Al_2O_3 with liquid water, and inhibits the transport of reactants/products involved with corrosion of the Al_2O_3 layer.

EXPERIMENTAL DETAILS

Interdigitated electrode (IDE) test structures were adapted to evaluate the performance of the bi-layer coating. IDEs were fabricated using standard lift-off lithographic techniques. 500-µm thick fused silica substrate was used due to its high resistivity [15]. Electrodes were 130 µm wide with the same spacing in between. The electrodes were composed of sputtered Ti(100 nm)/Pt(150 nm)/Au(150 nm) sequentially to match the metallization used for wireless version of Utah electrode arrays (UEAs)[16]. The IDEs were then annealed at 375 °C in Forming gas (98% of Ar and 2% of H_2) for 45 minutes (Figure 1). Two lead wires were soldered for later electrical measurements. Thin Al_2O_3 films were deposited using plasma assisted atomic layer deposition (PAALD) by sequentially exposing IDEs to Trimethylaluminum (TMA) vapor and oxygen plasma for 500 cycles at 120 °C using Fiji F200 (Cambridge NanoTech Inc.). The PAALD cycle consisted of 0.06 s TMA pulse, 10 s argon purge (200 SCCM), 20 s O_2 plasma (20 SCCM), and 5 s argon purge (200 SCCM) at 0.3 mTorr. PAALD process was preferred for its lower deposition temperature and shorter purge time comparing with a thermal ALD process. The deposition rate was about 1.04 Å/cycle on silicon substrate, measured by using VASE ellipsometer (J.A. Woollam Co., Inc), which is similar to Langereis *et al.* reported[12]. 6 µm of

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Parylene C were then deposited on top of Al_2O_3 using the standard Gorham CVD process [8] using LabTop 3000 Parylene coater (Para Tech Coating). Vapor phase Silane A-174 (Momentive Performance Materials) was used as adhesion promoter between Al_2O_3 and Parylene C. 6-ml vials filled with 1× phosphate buffered solution (PBS) were used to soak the bi-layer coated IDEs at temperatures from 37 °C to 80 °C. PBS was changed every other week to minimize the ion concentration change due to water evaporation. Leakage current and electrochemical impedance spectroscopy were used to assess the long-term insulation performance of the bi-layer encapsulation.

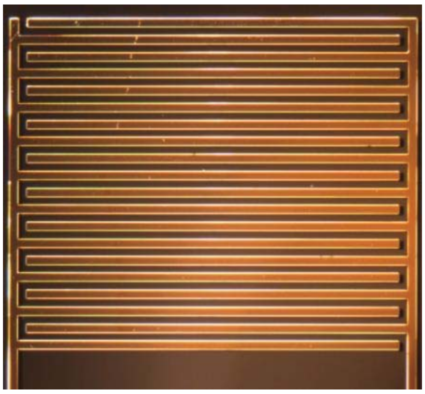

Figure 1: Micrograph of the fabricated interdigitated electrodes (IDEs).

RESULTS AND DISCUSSION

Film Characterization

AFM micrographs show that the as-deposited 52-nm Al_2O_3 film has RMS surface roughness of 0.48 nm, which is a slight increase from the substrate (RMS surface roughness of 0.17 nm). X-ray photoelectron spectroscopy was used to analyze the composition of as-deposited film with depth profiling. The measured results are presented in Table 1. The Al/O ratio was about 1.41 and no Ar gas was found in the film by XPS.

Table 1: XPS analysis of an Al_2O_3 layer deposited using PAALD process – 500 cycles of TMA+O_2 gas on silicon wafer.

Etch time (s)	O 1s	C 1s	Al_2 p	Si_2 p
0	51.5	14.5	34	0
300	57.3	0.5	42.2	0
600	58.5	0	41.5	0
900	57.8	0	41.9	0.3
1200	13.2	1.4	7.8	77.6
1500	0.9	0	0	99.1

Leakage Current

Leakage current is an important metric to evaluate the performance of the encapsulation. Leakage current measurement can be used to detect DC passes in the encapsulation created by pin holes and film defects. Leakage current was measured by applying 5 VDC to the two electrodes of the IDEs. Figure 2 presents leakage current for IDEs soaked at different temperatures. The leakage current was about 1 pA in air; then it immediately increased to about 20 pA after being soaked in PBS. The initial increase in leakage current indicated a decrease in DC resistance, due to shorter effective distance for resistance in PBS [17]. For Parylene C coated IDEs, the leakage current was over 1 nA after 60 days of soaking at 67 °C, indicating degradation and failure of the coating. However, for alumina and Parylene coated IDEs, the leakage current was constantly about 20 pA during 260 days of soak testing at 67 °C. Lower leakage current for a longer period of soaking time at the same temperature clearly shows alumina and Parylene C bi-layer coating is superior compared with Parylene C coating.

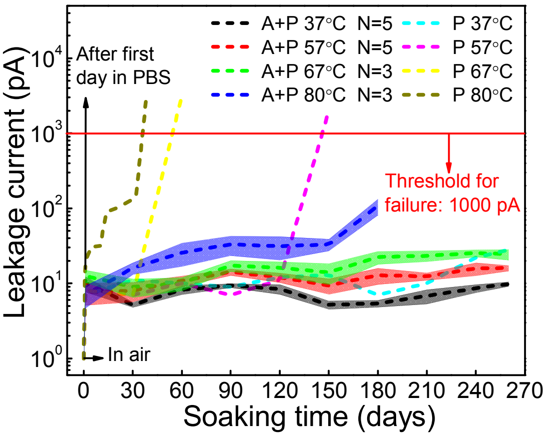

Figure 2: The leakage current from IDE test structures is plotted versus time over the 260-day test period. "A+P" denotes alumina and Parylene coating, "P" denotes Parylene coating, "N" denotes number of samples used in this plot. "Day 0" means samples were in air before soaking test.

Impedance

Impedance of encapsulation and its changes over time are very important since they are indications of insulation performance of the coating. Good insulation can reduce cross talks and signal loss via shunting with physiological environment, especially for implantable devices with active electronics. Electrochemical impedance spectroscopy (EIS) is widely used to evaluate the longevity and degradation kinetics of coating. The impedance was first measured in air from 1 Hz to 1 MHz, as shown in Figure 3. The impedance was almost a straight line with slope of -1 over the whole frequency range and the phase was about -88° during the 260 days of soak testing at 37 °C, indicating the bi-layer coated IDEs were purely capacitive. The long-term constant capacitive property of IDEs suggested the stability and minimal degradation of the bi-layer encapsulation. Impedance at 1 kHz is very important for biomedical applications, such as neural recording and stimulation, since the frequency of action potentials is about 1 kHz. After the initial drop due to media change from air to PBS, the impedance of bi-layer coated IDEs at 1 kHz is about 3.5 MΩ with a phase of -88° during the whole soaking period. This is about one order of magnitude higher than J. Hsu *et al.*[6] (IDEs with the same dimensions used) and J. Seymour *et al.*[7] reported by using Parylene C as encapsulation.

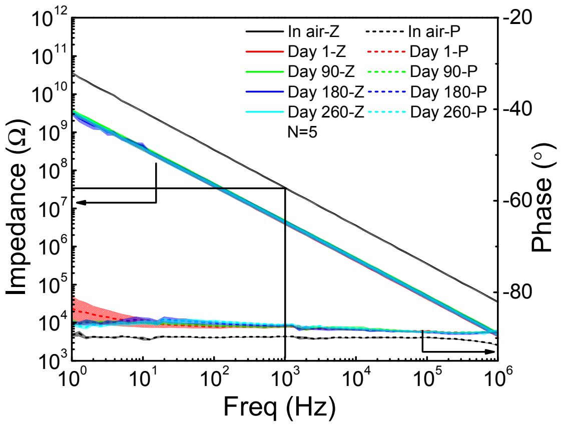

Figure 3: Bode plots of impedance spectroscopy of 260-day soaking test in 37 °C PBS for alumina and Parylene C encapsulation. The impedance is denoted by Z and the phase is denoted by P in the legend. Data was acquired from 5 samples (N=5) and shaded areas represent the standard error. There was an initial drop for the impedance from air to PBS; then the impedance and phased remained nearly constant.

Accelerated lifetime testing was used to expedite the validation process for this new encapsulation scheme as the films need to protect implantable devices for years or even decades for chronic implantation. Body temperature was chosen as a baseline and accelerated aging factor (F) was calculated (Table 2) based on a doubling of aging rate on every 10 °C increase[18, 19]. Impedance and phase of bi-layer coated IDEs soaked at different temperature were almost identical during the whole period, as presented in Figure 4. IDEs at higher temperature were expected to have lower impedance, higher phase and higher leakage current after the same period of soaking time based on Arrhenius equation. However, no significant difference has been observed in terms of leakage current (Figure 2) and impedance (Figure 4) for IDEs at 37 °C, 57 °C and 67 °C. Because of this, the accelerated lifetime was not able to resolve the characteristics of the encapsulation degradation at this time. For IDEs at 80 °C, the leakage current increased from 20 pA to 100 pA and phase started to increase at low frequency ranges. This indicated the beginning of degradation of the encapsulation.

Bias and Topography Effect

Biomedical implants with active electronics require power. Depending on the voltage and current characteristics, power supply can generate additional aging factor affecting the performance of the encapsulation [20, 21], which is different from temperature aging mechanism. Bias voltage effects were studied by applying 5 VDC continuous bias to Parylene coated and alumina and Parylene bi-layered coated IDEs during the whole soaking period. The continuous bias showed no effect on alumina and Parylene C coated samples yet. However, it reduced the lifetime of Parylene C coated samples by ~ 75% compared with those without continuous bias. This showed that continuous bias accelerated the failure process of Parylene coating while it has significant less or no effect on alumina and Parylene bi-layer coating.

Table 2: Accelerated aging factors and equivalent soaking time for different elevated temperatures relative to 37 °C.

Temperature (°C)	Aging factor (F)	Real soaking time (day)	Equivalent soaking time at 37 °C (day)
37	1	260	260
57	4	260	1040
67	8	260	2080
80	20	180	3600

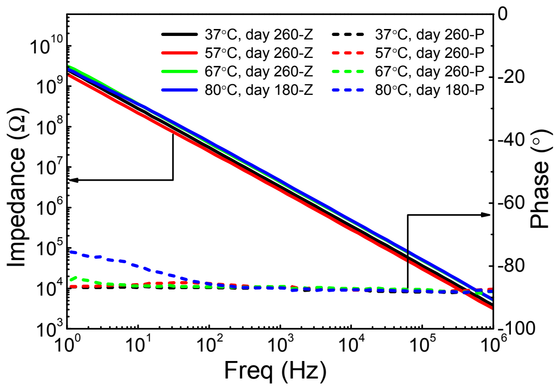

Figure 4: Impedance spectroscopy plots of IDEs with alumina and Parylene coating at 37 °C and elevated temperature for accelerated testing in PBS. The impedance is denoted by Z and the phase is denoted by P in the legend. All the samples are still under testing.

The encapsulation performance on planar IDEs might be different from real biomedical devices with complex topography. To simulate the real devices, hand-wound coils and SMD capacitors were added to the surface of planar IDEs to create the topography. The lifetime of alumina and Parylene coated IDEs with the extra topography was only about 50% of that of planar test structures. This suggested that topography significantly affected the performance of the encapsulation.

CONCLUSION

We have demonstrated that combination of ALD alumina and Parylene C bi-layer encapsulation had excellent insulation performance for IDE test structures. IDEs coated with Alumina and Parylene had stably high long-term insulation impedance of 3.5 MΩ at 1 kHz, low leakage current of 20 pA over 6 years of equivalent soaking period at 37 °C. Also, bi-layer coated IDE test structures showed better resistance to continuous bias voltage compared with Parylene only coated IDEs. All those made this novel bi-layer encapsulation very promising for chronic implantable devices.

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