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Effect of Signals on the Encapsulation Performance of Parylene Coated Platinum Tracks for Active Medical Implants

Kambiz Nanbakhsh, Marta Kluba, Barbara Pahl, Florian Bourgeois, Ronald Dekker, Wouter Serdijn, and Vasiliki Giagka

***Abstract*— Platinum is widely used as the electrode material for implantable devices. Owing to its high biostability and corrosion resistivity, platinum could also be used as the main metallization for tracks in active implants. Towards this goal, in this work we investigate the stability of parylene-coated Pt tracks using passive and active tests. The test samples in this study are Pt-on-SiO_2 interdigitated comb structures. During testing all samples were immersed in saline for 150 days; for passive testing, the samples were left unbiased, whilst for active testing, samples were exposed to two different stress signals: a 5 V DC and a 5 Vp 500 pulses per second biphasic signal. All samples were monitored over time using impedance spectroscopy combined with optical inspection. After the first two weeks of immersion, delamination spots were observed on the Pt tracks for both passive and actively tested samples. Despite the delamination spots, the unbiased samples maintained high impedances until the end of the study. For the actively stressed samples, two different failure mechanisms were observed which were signal related. DC stressed samples showed severe parylene cracking mainly due to the electrolysis of the condensed water. Biphasically stressed samples showed gradual Pt dissolution and migration. These results contribute to a better understanding of the failure mechanisms of Pt tracks in active implants and suggest that new testing paradigms may be necessary to fully assess the long-term reliability of these devices.**

I. Introduction

For implantable medical devices, life-time reliability has always been a major concern when introducing novel designs to the market. Degradation of metal tracks is a common failure mechanism severely affecting reliability. In active implants, i.e. implants with embedded electronics, tracks are the metallization used for electrically connecting different components to each other (Fig. 1). Allowing the metal to degrade will result in an increased track resistance, cause a short between two adjacent tracks or a hard open.

Conventionally, medical devices have relied on a titanium (Ti) package to protect the inside metal tracks and electronics against the ionic fluids in the body. Driven by miniaturization, especially arising from the emerging field of bioelectronic medicine, in recent years great improvements have been made in polymeric packaging whereby soft, light weight and flexible implants are be realized [1-2].

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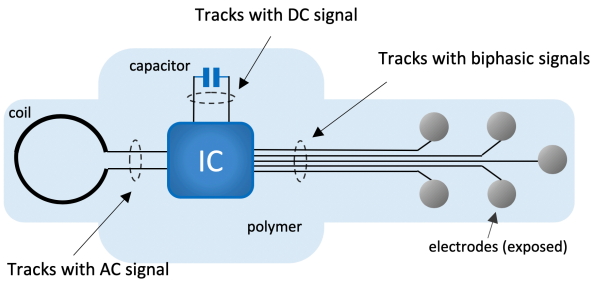


Figure 1: System overview of a wirelessly powered active implant.

The difference in nature of polymer to Ti packages, however, has required dedicated tests to be devised in order to assess the lifetime reliability of polymer coated metal tracks in wet environments. Within the aqueous and ionic environment of the body, the common degradation scenarios for metals are chemical corrosion, inter-metallic corrosion and electrochemical migration (ECM) [2]. For all these scenarios, the presence of liquid water is necessary. Yet, for the latter case, the presence of an electric field between the metals is the main driving force [2-4].

Within an implant (Fig. 1), the metal tracks would be carrying various signals essential for the functionality of the device: alternating current (AC) signals from the coil, direct current (DC) signals between the integrated circuit (IC) and the energy storage capacitor and biphasic signals intended for tissue stimulation (generated by the IC and delivered to the tissue via the electrodes).

There have been already studies investigating the effect of DC stress signals on the long-term reliability of polymer-encapsulated metal tracks. In [3], for example, the authors investigated the lifetime encapsulation of epoxy coated gold (Au) metallization when applied to a 12 V DC signal and in [4] the authors have reported on the effects of a continuous 5 V DC signal in reducing the encapsulation performance of parylene coated Au tracks.

In this study we intend to investigate the stability of parylene encapsulated platinum (Pt) tracks in the presence of DC and biphasic stress signals. Pt is commonly used as the material for interface electrodes [3], however, owing to its high biocompatibility and corrosion resistivity, it is also often used as the main metallization in implants [5-6]. Using the same materials for the electrodes and tracks would enable their fabrication to be done in one processing step, simplifying the fabrication flow. Furthermore, such an approach would minimize any inter-metallic junctions which otherwise could be a potential failure point.

For this study, test samples with Pt metallization have been fabricated and coated using a parylene layer. Section II presents the detailed sample design and coating process. The experimental set-up is also given in this section. The study results together with a discussion are given in section III. Finally, the conclusions are drawn in section IV.

II. Experimental Details

A. Sample Fabrication

1) Metallization

To investigate the stability of encapsulated Pt tracks, interdigitated comb (IDC) structures have been used. These structures are widely used as test vehicles for evaluating the encapsulation performance because of their simplicity and high sensitivity [3-4]. Moreover, by using these structures an electric field could be applied between the two metal structures, mimicking real use conditions. For this work, we have designed and fabricated silicon-based test devices with Pt metallization. First, a 1 µm thick plasma enhanced chemical vapor deposition (PECVD) SiO_2 layer was deposited on a 675 µm thick 6-inch silicon substrate. Next, a 10 nm Ti adhesion layer and 600 nm of Pt were sputter deposited on the whole wafer in one deposition process - without breaking the vacuum. The metallization layer was patterned in a lithographic process using a positive photoresist mask and subsequent ion-milling of the Pt and Ti. During that process the IDCs, bond pads for wire connection and interconnects were defined. Finally, the wafer was diced into individual dice (see Fig. 2a). The 600 nm thick layer of Pt enables direct wire attachment to the bond pad without a need of applying an additional layer of metallization. To shield the comb structures from external electrical disturbances, the interdigitated combs are surrounded with a guard ring also finished with a bond pad. The guard ring and the bond pads are located well within the elongated silicon chip (> 0.8 mm from the die edge) to prevent possible damage of the structures during handing and to eliminate risk of device failure due to delamination progressing form the devices edge towards the comb structures.

2) Parylene-C Coating

Prior to parylene processing, the samples were cleaned in acetone, isopropanol (IPA) and deionized water (DI). Parylene was deposited at Comelec using their C30S system at room temperature. The system includes a two-minute O_2 plasma step followed by an adhesion promoter processing step. For the adhesion promoter, thiol functional groups were used. After conformal parylene deposition, an annealing step was performed at 140 °C under 200 mbar of nitrogen (N_2) for two hours. Fig. 2b schematically illustrates the cross section of the fabricated samples.

B. Experimental Set-up and Stress Voltages

For soak testing, 50 ml vials were used which were filled with 1X phosphate buffered saline (PBS) with a composition of 0.0027 M KCl and 0.0137 M NaCl with pH of 7.4.

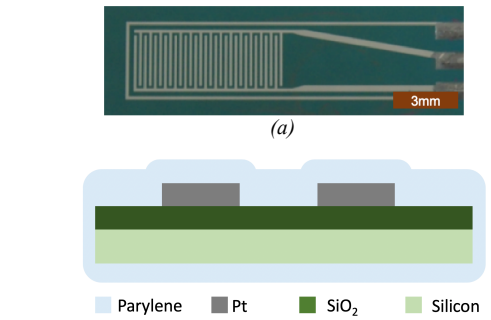


Figure 2: (a) IDC used as test vehicle, (b) cross section representation of layers.

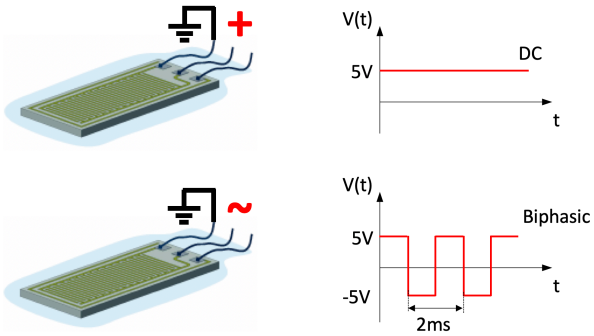


Figure 3: Voltage signals applied to IDC test structures.

The soaking tests were performed in a temperature-controlled water bath. Proper care on the vial sealing was taken to minimize water evaporation. More details on the set-up specifications and preparation can be found in [7]. Three samples were used for passive testing where no signals were applied to the samples except for the measurement signal, applied once every two weeks. These samples will be used as the control group for later comparison. For active testing, three samples have been connected to a 5 V DC signal and another three to a 5 Vp 500 pulses per second (pps) biphasic signal. Fig. 3 illustrates the waveform, amplitude and pulse repetition rate of the two different signals used for this study. The soaking started at room temperature (22 °C), at day 30 was increased to 37 °C and then later at day 100 was increased to 67 °C. The incremental increase was used to see possible acceleration due to temperature increase.

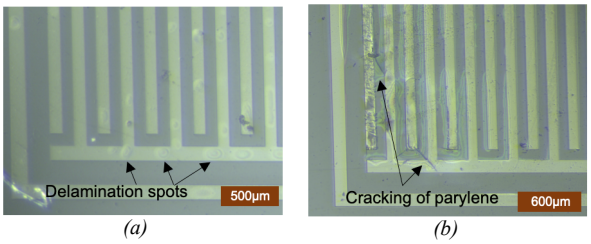
C. Measurement Method

For this work, electrochemical impedance spectroscopy (EIS) experiments were carried out using a Solartron potentiostat in combination with a frequency response analyzer (FRA). Two electrode EIS measurements are made in the frequency range of 100 mHz to 100 KHz using a 50 mV RMS sinusoidal signal. A low amplitude signal has been chosen to minimize the effects from the measurement signal on the samples. During measurement, the bias voltage was set to zero between the working and counter electrodes.

III. Results and Discussion

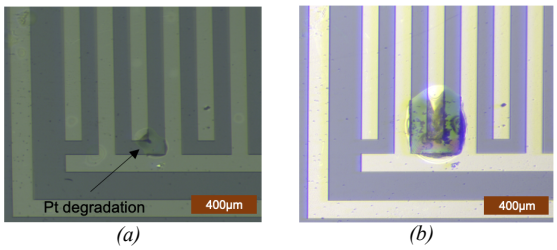
A. Optical Inspection

For optical inspection, all samples were taken out of PBS, rinsed with deionized water (DI), blow-dried and optically inspected using a microscope once every two weeks. The first inspections after 14 days of soaking at room temperature revealed the presence of Newton rings (not visible prior to the beginning of the experiment) on the Pt tracks for all samples. Fig. 4a shows the formation of these Newton rings on a non-stressed sample. The Newton rings were spread randomly on the Pt traces and mostly present where the parylene-metal interface was larger. These rings are created due to the local delamination of the parylene from Pt which would later yield to water condensation. Further inspections, however, revealed that the delamination spots were confined to the Pt metallization only, suggesting the good adhesion of parylene to SiO_2. Increasing the temperature from 22 °C to 67 °C further increased the number of delamination spots on the metal tracks. For the non-stressed samples, despite the nucleation of water on the metallization, no signs of metal degradation was found. For the actively stressed samples, however, two different failure mechanisms were observed which were stress signal related. For the 5 V DC samples, severe cracking of the parylene was found (Fig. 4b). All the samples stressed by DC showed cracking of the coating layer, exposing the metal to PBS. Failure for DC stressed samples occurred at different times between day 14 (T=22 °C) and day 130 (T=67 °C). Samples stressed using the biphasic signal, on the other hand, started showing signs of color change on the Pt tracks after 80 days. This was accompanied by a delaminated area between the metal combs. Over time, the delamination areas between the metal tracks grew and features were seen under the parylene layer, possibly signs of severe Pt degradation (Fig. 5). Increasing the temperature from 37 °C to 67 °C accelerated the degradation process and the area showed bulging of the parylene layer. This was observed on all the three samples stressed by the 5 Vp biphasic signal.



(a) (b)

Figure 4: (a) Delamination spots visible on Pt tracks, (b) cracking of parylene layer due to water electrolysis.



(a) (b)

Figure 5: Sample stressed with a biphasic signal, (a) day=135, (b) day=145.

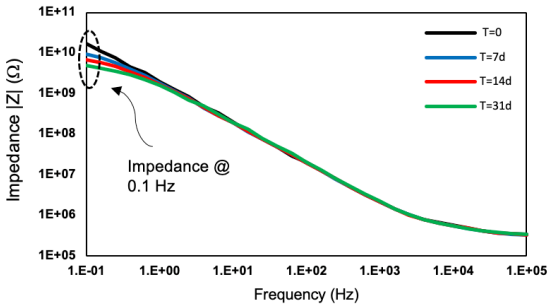


Figure 6: EIS results for a biphasically stressed sample over a period of one month soaking.

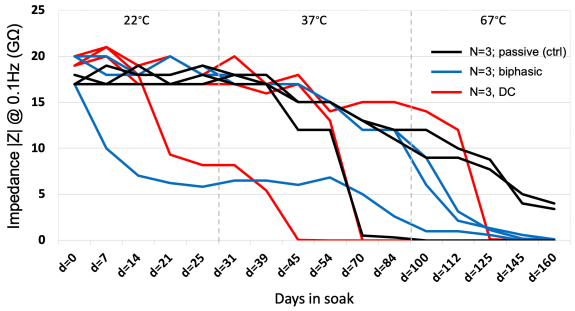


Figure 7: Impedance @ 0.1Hz over time for passive and actively stressed samples.

B. Measurements Results

To detect changes between the metal tracks on the IDC, weekly EIS measurements were done. Change is considered any deviation from the first EIS done immediately after soaking the sample in PBS. Fig. 6 shows the results extracted from EIS measurements for a sample stressed with a biphasic signal over a period of 30 days at 22 °C. It can be seen that changes in the impedance are mostly occurring in the lower frequency range (<1Hz) indicating possible water condensation between the tracks. Higher frequencies (> 50 KHz) are usually representing the capacitance of the combs and the routings to the sample. For this reason, for all samples, the impedance (|Z|) at 0.1 Hz was taken as an indicator to monitor changes over time. Any impedance drop below 100 MΩ is considered a failure. Fig. 7 shows the EIS results @ 0.1 Hz for all samples over the duration of the study. As seen in this figure, impedance for all samples drop as the temperature increases. This could be a result of more water penetration and condensation between the comb structures on the IDC. Additionally, temperature could also accelerate the electrochemical reactions taking place between the metal tracks for the actively stressed samples, thereby, increasing the conductivity of the confined water between the metallization [2]-[4]. This could explain the

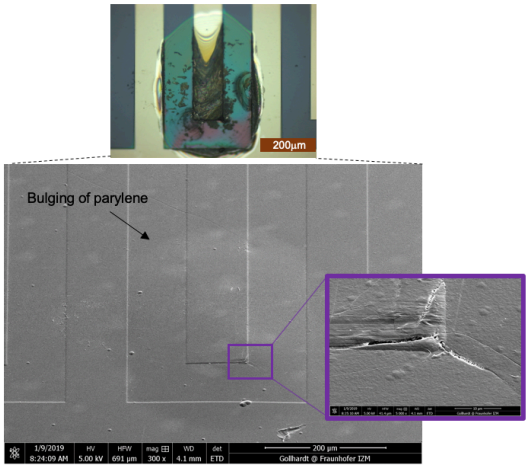


Figure 8: SEM image showing a bulging and cracking on the parylene layer.

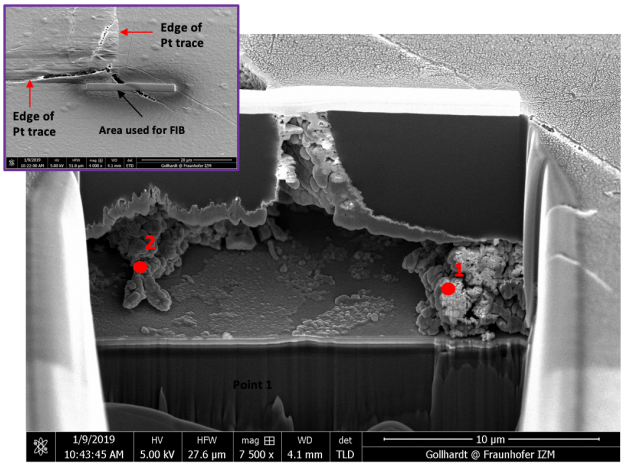


Figure 9: FIB and EDX analysis showing Pt dissolution and migration under the parylene layer.

relatively lower drop in impedance for non-stressed samples. For one of the non-stressed samples, however, the change in impedance occurred sooner with respect to the other two. Closer examination, revealed that the drop in impedance was due to water condensation in the connections to the sample and not on the IDC itself.

C. SEM and EDS analysis

To further investigate the failure mode for the biphasically stressed samples, scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) was used. Fig. 8 shows the SEM results of the area shown in Fig 5b. Bulging is seen in the middle section of Fig. 8 which is due to delamination and water nucleation under the parylene layer. Closer examination also showed a crack forming in the parylene layer. This crack could have been initially present in the layer or it could have formed due to the stress build up from the bulging. By examining the EIS results for this sample, however, we understand that the crack could not have been initially present due to the high impedance values measured on this sample for the first weeks of soaking. To analyze the features forming under the parylene layer, focused ion beam (FIB) was used to cut through the layer. The top left corner of Fig. 9 shows the area next to the Pt tracks which was used for FIB and EDS. Fig. 9 shows the growing features seen under the parylene layer as a result of the biphasic stress signal. EDS results for the two points selected in Fig. 9 showed traces of Pt and chlorine (Cl), indicating platinum degradation in the form of dissolution and migration. A second FIB and EDS analysis was done on a different location of the sample showing similar features. Results also revealed traces of Pt and chlorine in the growing features between the metal tracks.

D. Discussion

As reported in literature, parylene, similar to all polymers, is permeable to moisture [2]-[8]. The weak adhesion of parylene to Pt, therefore, would allow water condensation on the Pt tracks. As the optical inspection and EIS results showed, however, for the unbiased (passive) samples this did not result in Pt degradation. On the other hand, the good adhesion of parylene to SiO_2 prevented water condensation between two neighboring metal tracks. As the EIS measurements in section III.B showed, when the parylene-SiO_2 interface holds, high impedances (> 10 GΩ) can be achieved. Over time, however, as the size of the water droplet on the Pt tracks increases, more stress will be introduced on the parylene-SiO_2 interface. Together with the capillary forces of water and the possible ionic contamination on the SiO_2 surface, the delamination will spread further into the parylene-SiO_2 interface. Once a water channel is formed between two adjacent tracks, the passing DC current would result in electrolysis and the resulting pressure build-up due to gas evolution would eventually crack the parylene and expose the metal. For samples applied to the biphasic signal, however, a different mechanism lead to failure. As a water channel was formed between two Pt tracks, the biphasic stress signal initiated an electrochemical reaction leading to the degradation of Pt. The degradation of Pt during potential cycling has been studied by others [9], [10], but primarily for Pt electrodes exposed to acidic media, i.e. the Pt surface was fully exposed to the solution. In these studies, it has been shown that for Pt to dissolve and migrate under biphasic voltages, the presence of oxygen and chloride (Cl^-) ions is needed. In the context of encapsulated Pt tracks, however, it is surprising to see Pt dissolution and migration, given the limited supply of Cl^- and oxygen under the polymer.

It is beyond the scope of this paper to analyze the dissolution process of Pt, yet it is important to understand how the dissolution started and why it resulted in gradual bulging of the parylene layer. It is known from literature [1-4] that all polymers, similar to moisture, are permeable to oxygen while being good barriers against ions. It is unclear, therefore, how the necessary chloride ions for the dissolution

process were present at the parylene-SiO_2/Pt interface. Two hypotheses could be given for this. First, despite the rigorous cleaning, the chloride ions could be present as surface contamination. The second hypothesis is that the chloride ions, in a limited scale, penetrated through the parylene layer and initiated the dissolution process.

For both failure mechanisms reported here, the poor adhesion of parylene to Pt together with the high water vapor transmission rate (WVTR) of parylene were the main causes of failure. With this motivation, a new hybrid process is being developed at Comelec that would enable a multilayer deposition of parylene and inorganic layers. The resulting coating, therefore, would be a multilayer with much higher barrier properties than a single parylene layer. This process is all done in one chamber without vacuum break which is essential for keeping the contamination low.

Nevertheless, the results reported here suggest that the design of testing/evaluation strategies aiming to assess the long-term reliability of active implants would have to accommodate a variety of stress signals to fully investigate the various scenarios that can occur during the implants lifetime operation.

IV. Conclusion

In conclusion, in this work we investigate the stability of parylene encapsulated Pt tracks when subjected to a DC and biphasic stress signal. Results suggest two different failure mechanism for the two different stress signals. The DC signal caused electrolysis of the condensed water resulting in parylene cracking while for the samples stressed with the biphasic signal, Pt dissolution and migration was found to be the main failure mechanism. The root cause for both failures were the poor adhesion of parylene to Pt and the high WVTR of parylene. The above results contribute to our understanding of the failure mechanisms of Pt tracks on active implants, and suggest that new testing paradigms may be necessary to fully assess the long-term reliability of these devices.

Acknowledgments

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References

[1] V. Giagka and W. A. Serdijn, "Realizing flexible bioelectronic medicines for accessing the peripheral nerves – technology considerations," *Bioelectron. Med.*, vol. 4, no. 8, pp. 1–10, 2018.

[2] A. Vanhoestenberghe, N. Donaldson, "Corrosion of silicon integrated circuits and lifetime predictions in implantable electronic devices," *J. Neural Eng.* 10(3), 031002, 2013.

[3] X. Huang, P. M. Denprasert, , L. Zhou, A. N. Vest, S. Kohan and G. E. Loeb, "Accelerated life-test methods and results for implantable electronic devices with adhesive encapsulation," *Biomedical Microdevices*, 19(3), 46-57, 2017.

[4] X. Xie, L. Rieth, R. Caldwell, M. Diwekar, P. Tathireddy, R. Sharma, F. Solzbacher, "Long-term bilayer encapsulation performance of atomic layer deposited Al2O3 and parylene C for biomedical implantable devices," *IEEE Trans. Biomed. Eng.* 60, 2943–2951, 2013.

[5] M. Schuettler, S. Stiess, B. V. King, and G. J. Suaning, "Fabrication of implantable microelectrode arrays by laser cutting of silicone rubber and platinum foil," *J. Neural Eng*., vol. 2, no. 1, pp. S121 – S128, Feb. 2005.

[6] V. Giagka, A. Demosthenous, and N. Donaldson, "Flexible active electrode arrays with ASICs that fit inside the rat's spinal canal," *Biomed. Microdev.*, vol. 17, no. 6, pp. 106 – 118, Dec. 2015.

[7] N. Donaldson, C. Lamont, A. Shah Idil, M. Mentink, and T. Perkins, "Apparatus to investigate the insulation impedance and accelerated life- testing of neural interfaces," *J. Neural Eng,* 2018. [Online]. Available: http://iopscience.iop.org/article/10.1088/1741 2552/aadeac

[8] J. Ortigoza-Diaz, K. Scholten, C. Larson, A. Cobo, T. Hudson, J. Yoo, A. Baldwin, A. Weltman Hirschberg, E. Meng, "Techniques and Considerations in the Microfabrication of Parylene C Microelectromechanical Systems," *Micromachines,* 2018, 9, 422.

[9] S. Geiger, S. Cherevko, K.J.J.J. Mayrhofer, "Dissolution of platinum in presence of chloride traces," *Electrochim. Acta,* 2015, 179, 24–31.

[10] Z. Wang, E. Tada, A. Nishikata, "In Situ Analysis of Chloride Effect on Platinum Dissolution by a Channel Flow Multi-Electrode System," *J. Electrochem.* Soc2014, 161, F845−F849.